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Preservation of Reactor Test Unit and Desulfurization of Gob Pile Samples

TRW Defense and Space Systems Group, Redondo Beach, CA

Prepared for

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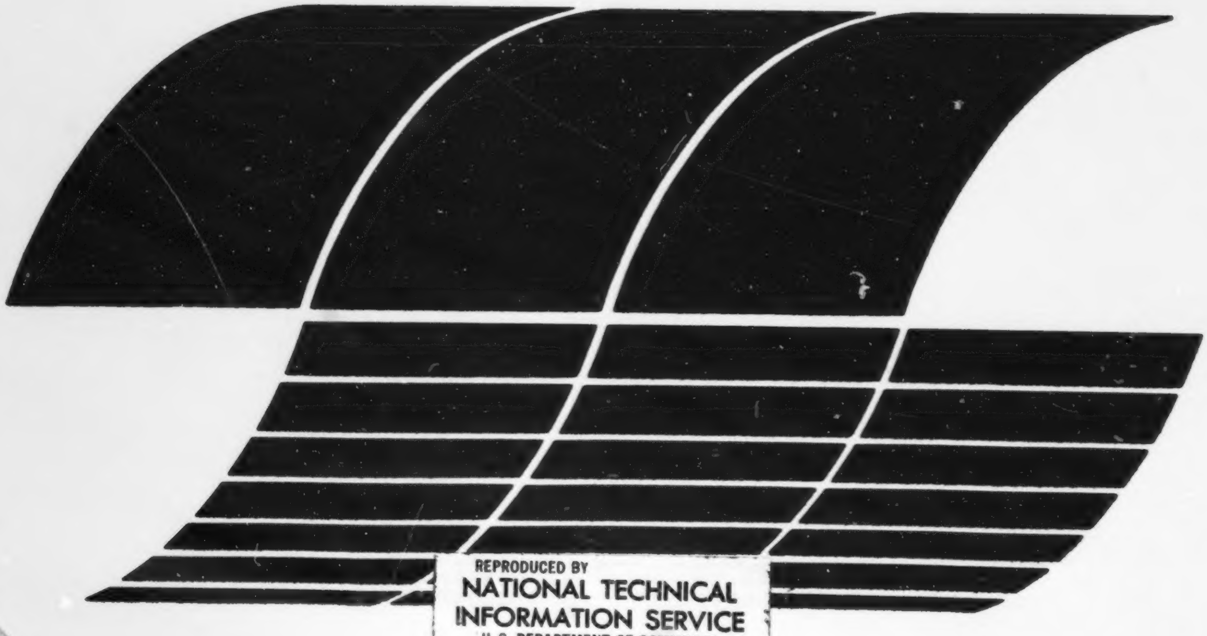

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16. ABSTRACT

The report gives results of preservation and desulfurization studies associated with the Reactor Test Unit (RTU), an 8-ton per day test plant used in the EPA-sponsored development of the Meyers Process for ferric sulfate leaching of pyritic sulfur from coal. RTU operation in 1977 and 1978 showed that it could be run continuously on three shifts to reduce the sulfur content of the feed coal to meet the 1.2 lb SOx Emission Standard for New Stationary Sources. Corrosion was encountered in the stainless steel main reactor vessel which required modification prior to further testing. The present program provides a complete corrosion assessment, established specifications for a tail-end elemental sulfur extraction unit, and developed maintenance and upkeep requirements for the RTU. A Meyers Process modification, involving a preliminary float and sink operation in the ferric sulfate leach solution, followed by Meyers processing of the sink (high pyrite) fraction was investigated at bench scale. Experimental verification and applicability assessment of this new approach was performed on both coal wastes and Eastern Interior Basin run-of-mine coal.

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ABSTRACT

The development of the Meyers Process for ferric sulfate leaching of pyritic sulfur from coal has been sponsored through construction and operation of an eight ton per day test plant, termed the Reactor Test Unit (RTU). Operation of the test plant in 1977 and 1978 showed that the unit could be run continuously in three-shift operation to reduce the sulfur content of the feed coal to meet the 1.2 pound Standard for New Stationary Sources. Corrosion problems were encountered in the stainless steel main reactor which required modifications prior to any further testing. This present program provided a complete corrosion assessment, specification of a new main reactor of titanium, specification of a tail-end elemental sulfur extraction unit and maintenance of the RTU.

A modification of the Meyers Process, involving a preliminary float and sink operation in the ferric sulfate leach solution, followed by Meyers processing of the sink (high pyrite) fraction was investigated at bench-scale. Experimental verification and applicability assessment of this new approach was performed on both waste and Eastern Interior Basin run-of-mine coal.

CONTENTS

Abstract iii
Figures vi
Tables xi
1.0 Introduction. 1
2.0 Conclusions 6
3.0 Recommendations 7
4.0 Reactor Test Unit Inspection and Preservation 8
  4.1 Introduction 8
  4.2 Background 8
  4.3 Routine Maintenance During Standby 11
  4.4 RTU Inspection and Inspection Results. 16
    4.4.1 Coal Feed System. 23
    4.4.2 Leach Solution and Waste Liquid Storage System. 24
    4.4.3 Leach Solution Feed System. 24
    4.4.4 Coal Slurry Preparation System. 27
    4.4.5 Primary Reactor System. 30
    4.4.6 Gas Vent System 31
    4.4.7 Secondary Reactor System. 32
    4.4.8 Filtration and Leach Solution Recovery System . 33
    4.4.9 Gasket Sealing Surfaces 35
    4.4.10 Equipment Spare Parts 38
5.0 Bench-Scale Extractions (Task 2A) 124
  5.1 TVA Coal Studies (Task 2A1) 124
    5.1.1 Float-Sink (Organic Liquid) (Task 2A1a) 124
    5.1.2 Float-Sink (Leach Solution) (Task 2A1b) 126
    5.1.3 Meyers Process on ROM Coal (Sink) (Task 2A1c) . 126
    5.1.4 Float-Sink 3/8" x 0 Coal (Task 2A1d) 126

CONTENTS (Continued)

5.2 Gob Pile Coals (Task 2A2) 134

5.2.1 Float-Sink - Gob Coals (Task 2A2) 134

5.2.2 Float-Sink Analysis (Task 2A2). 136

5.2.3 Meyers Processing (Task 2A2). 139

6.0 Specifications and Estimates for R-1 Reactor Replacement. . . 143

7.0 RTU Acetone Extraction Unit 148

7.1 TEU Conceptual Design. 148

7.1.1 Process Description 149

7.1.2 TEU Design Basis. 158

7.2 TEU Cost Estimate 158

8.0 Application Studies 166

8.1 Applicability of Gravichem Process for U.S. Coals. . . . 166

8.2 Applicability of the Gravichem Process for Waste Coal Recovery 168

8.2.1 Coal Oil or Coal Water Mixtures for Boiler Retrofit. 168

8.2.2 Gravifloat Process for Recovery of High Grade Fine Coal from Slurry Ponds 170

8.2.3 Waste Coal Recovery from TVA Mine 173

9.0 References 175

FIGURES

Number Page

1 Federal Energy Technology Test Facility Sponsored by U.S. Environmental Protection Agency at TRW's San Juan Capistrano Test Site. 2

2 Gravichem Process 4

3 RTU Schematic 10

4 RTU Control Console. 39

5 View RTU - West Side 40

6 Coal Bin Tilter and Oxygen Analyzer. 41

7 Coal Bin Tilter. 42

8 Equipment on Level 4 of RTU. 43

9 Vent Gas Scrubber T-4 and Knockout Drum V-1. 44

10 Coal Feed Tanks T-1 and T-6 and Weigh Belt Feeder A-3. 45

11 Slurry Mix Tank T-2. 46

12 Primary Reactor R-1. 47

13 Slurry Mix Tank T-2 and Primary Reactor R-1. 48

14 Belt Filter. 49

15 Filtrate Collection Equipment. 50

16 Recirculation Pumps for Reactor R-1. 51

17 Leach Solution Storage Tanks and Pumps 52

18 Rubber Boot Between Coal Storage Tank T-1 and Live Bottom Feeder A-2. 53

19 Rotary Vane from Coal Feed Valve A-4 54

20 Impeller Back Plate from Leach Solution Circulation Pump P-12. 55

FIGURES (Continued)

Number Page

21 Rotor from Leach Solution Feed Pump P-13. 56

22 Shaft Pins from Leach Solution Feed Pump P-13 57

23 Bubble Cap Tray Inside Knockout Drum V-1. 58

24 Weir Between Cells 1 and 2 of Mix Tank T-2. 59

25 Weir Between Cells 2 and 3 of Mix Tank T-2. 60

26 Agitator Blade from Cell 2 of Mix Tank T-2. 61

27 Thermocouple Probe TE-21 from Cell 3 of Mix Tank T-2. 62

28 Thermocouple Well Flange in Cell 3 of Mix Tank T-2. 63

29 Rotor from Slurry Feed Pump P-1 64

30 Rotor End from Slurry Feed Pump P-7 65

31 Rotor Center from Slurry Feed Pump P-7 66

32 Intermediate Drive Shaft from Slurry Feed Pump P-7. 67

33 Universal Linkage Shaft from Slurry Feed Pump P-7 68

34 Shaft Pins from Slurry Feed Pump P-7. 69

35 Retaining Ring from Slurry Feed Pump P-7. 70

36 Pipe Flange which Mates with Slurry Feed Pump P-1 Discharge Port 71

37 Weir and Vessel Wall Inside Cell 4 of Primary Reactor R-1 . . 72

38 Flange from Recirculation Loop for Cell 4 of Primary Reactor R-1 73

39 Valve Body from Recirculation Loop for Cell 4 of Primary Reactor R-1 74

40 Thermocouple Probe from Cell 5 of Primary Reactor R-1 75

41 Inlet to Recirculation Pump P-5 76

42 Outlet of Recirculation Pump P-5. 77

43 Pump Housing from Recirculation Pump P-5. 78

FIGURES (Continued)

Number Page

44 Impeller Back Plate from Recirculation Pump P-5 - Side View. 79

45 Impeller Back Plate from Recirculation Pump P-5 - End View . 80

46 Impeller from Recirculation Pump P-5 - Front View 81

47 Impeller from Recirculation Pump P-5 - Back View 82

48 Drive Shaft from Recirculation Pump P-5. 83

49 Float Well for Level Monitor Gauge LT-58 from Cell 5 of Primary Reactor R-1 84

50 Interior View of Secondary Reactor R-1 85

51 Inlet Flange Face of Leach Solution Circulation Pump P-12. . 86

52 Outlet Flange Face on Leach Solution Circulation Pump P-12 . 87

53 Flange Face in Inlet Isolation Valve T4-28 to Tank T-9 . . . 88

54 Flange Face on Reactor R-2 Manway. 89

55 Flange Face on Inlet Spool to Leach Solution Pump P-13 . . . 90

56 Flange Face from Leach Solution Feed Line to Foam Scrubber T-3. 91

57 Flange Face from Leach Solution Feed Line to Pump P-13 . . . 92

58 Flange Face on Basket Strainer SP-11 Outlet Port 93

59 Dissassemble Valve T4-6 from Filter Cake Wash Line to Tank T-9 94

60 Level Switch LSL-37 from Knockout Drum V-1 95

61 Lower Flange Face on Level Controller LC-39 for Knockout Drum V-1 96

62 Lower Flange Face on Knockout Drum V-1 97

63 Thermocouple Probe TE-148 from Knockout Drum V-1 98

64 Mating Flange to Thermocouple Probe TE-148. 99

65 Flange Face at Outlet of Heat Exchanger E-2. 100

FIGURES (Continued)

Number Page

66 Flange Face on Process Line Mating with Heat Exchanger E-2 Outlet. 101

67 Flange Face on Manway Port on Cell 2 of Mix Tank T-2. 102

68 Thermocouple Probe TE-21 from Cell 3 of Mix Tank T-2. 103

69 Flange Face on Thermocouple Probe Port on Cell 3 of Mix Tank T-2 104

70 Flange Face on Outlet of Slurry Feed Pump P-1 105

71 Flange Face on Outlet of Slurry Feed Pump P-7 106

72 Flange Face from Slurry Feed Line to Cell 3 of Primary Reactor R-1 107

73 Manway Cover to Cell 3 of Primary Reactor R-1 108

74 Face of Flange Mating with Low Level Control Valve LV-59. . . 109

75 Flange Face on Inlet of Low Level Control Valve LV-59 110

76 Flange Face on Outlet of Low Level Control Valve LV-59 . . . 111

77 Flange Face on Spool Separating Low Level Control Valve LV-59 and Level Control KV-241. 112

78 Flange Face on Inlet of Level Control Valve KV-241. 113

79 Flange Face on Outlet of Level Control Valve KV-241 114

80 Flange Face on Reactor R-1 Cell 4 Discharge Line to Disposal Tank T-9. 115

81 Flange Face on Valve R4-52 in Cell 4 Recirculation Loop for Reactor R-1 116

82 Seal Retainer Rings and Ball for Valve R4-50 from Cell 4 Recirculation Loop for Reactor R-1 117

83 Thermocouple Probe TE-56 from Cell 5 of Reactor R-1 118

84 Flange Gaskets from Heat Exchanger E-2 119

85 Flange Gaskets from Low Level Control Valve LV-59 120

86 Flange Gasket from Reactor R-1 Cell 4 Sample Valve R4-76 . . 121

FIGURES (Continued)

Number		Page
87	Flange Gaskets from Steam Inlet Line to Cell 1 of Reactor R-1 .	122
88	Flange Gasket from Filtrate Receiver V-2 Discharge Line. . . .	123
89	Gravichem Processing of TVA Coal	130
90	Size Distribution by Sieve Analysis of TVA Sink Coal	131
91	Pyritic Sulfur Leaching Data from 1.3 S.G. TVA Sink Coal (102°C Ambient Pressure)	133
92	Reactor Drawing	144
93	Process Flow Diagram	150
94	TEU Plot Plan.	154
95	TEU Front Elevation.	155
96	Tail End Unit Sketch	156
97	Gravifloat Processing of an Ohio Waste Coal.	171

TABLES

Number Page

1 Test Plant Data Taken Over 5-Day Period. 3

2 Specific RTU Shutdown Activities 13-16

3 RTU Preventive Maintenance Utilized During Shutdown. . . . 17-18

4 RTU Inspection Items 19-22

5 Settling Tank Float/Sink Separation/Analyses of TVA (As Received) Coal 125

6 Particle Size of Float and Sink Fractions of TVA Coal. . . 127

7 Float/Sink Separation/Analyses of TVA Coal 128

8 Rate Data on Processing 1.3 S.G. TVA Coal Sink Fraction with 7.5% Iron Solution (4% H_2SO_4) at 102°C and Atmospheric Pressure 129

9 Gob Pile Coal Samples. 134

10 Gravi-Separation of Banner Gob Coal. 135

11 Settling Tank Float/Sink Separation/Analyses of Gob Pile Coals (14 Mesh). 137

12 Settling Tank Float/Sink Separation/Analyses of Gob Pile Coals Peabody #2 Coal (As-Received) 138

13 Processing on Gob Piles Sink Fraction* @ 102°C (Ambient Pressure) 140

14 Processing of Peabody #2 Sink Fraction* @ 102°C (Ambient Pressure) 142

15 Design Notes 145

16 Fabrication Notes. 146

17 Reactor Bid Summary. 147

18 Acetone Extraction Principal Mass and Volumetric Flows . . 151-152

TABLES (Continued)

Number Page

19 Utility Requirements. 153

20 TEU Equipment Design Basis Summary. 159-162

21 Equipment List and FOB COST*. 163-164

22 Estimated TEU Construction Costs. 165

23 Potential Sulfur Emissions of Coal Treated by Gravichem[a] Process Compared with Deep Cleaning[b] for High Sulfur Coal Regions of U.S. 167

24 Sulfur Emissions Reduction Potential of Gravichem Process Compared with Deep Cleaning 169

25 Gravifloat Coal Oil Slurry. 170

26 Gravifloat Process Results* 172

1.0 INTRODUCTION

The development of the Meyers Process for ferric sulfate leaching of pyrite sulfur from coal has been sponsored by the U.S. Environmental Protection Agency through construction and operation of an eight ton per day test plant (Figure 1) termed the Reactor Test Unit (RTU), at TRW's San Juan Capistrano Test Site. This sponsorship was due to a desire to evaluate a backup sulfur oxides emission control system for front-end control which would be especially applicable to the small utilities and inudstrial boilers. The Meyers Process removes 95% of the pyritic sulfur from coal leaving essentially only organic sulfur in the product. An extensive laboratory test survey of U.S. coals showed that the process could reduce the sulfur content of 1/3 of Appalachian coal to meet the New Source Performance Standards then established by the Environmental Protection Agency of 1.2 lbs $SO_2/10^6$ Btu.

Operation of the test plant in 1977 and 1978 on an Appalachian coal, supplied by the American Electric Power Service Corporation, showed that the unit could be run continuously in three-shift operation to reduce the sulfur content of the coal to meet the 1.2 standard (Table 1). Corrosion problems were encountered in the main reactor which required modifications prior to any further testing. Plant operation was then suspended and this contract was awarded to provide corrosion assessment, specification of a new main reactor, specification of a tail-end unit and maintenance of the plant.

During the performance of the RTU project, the Gravichem modification of the Meyers Process (Figure 2) was conceived in which coal is first allowed to float and sink in the ferric sulfate leach solution, the float product contains very little pyrite and is washed free of iron sulfate and is ready for use, while only the sink fraction (in which the pyrite is concentrated) is taken through the Meyers Process for removal of pyritic sulfur.

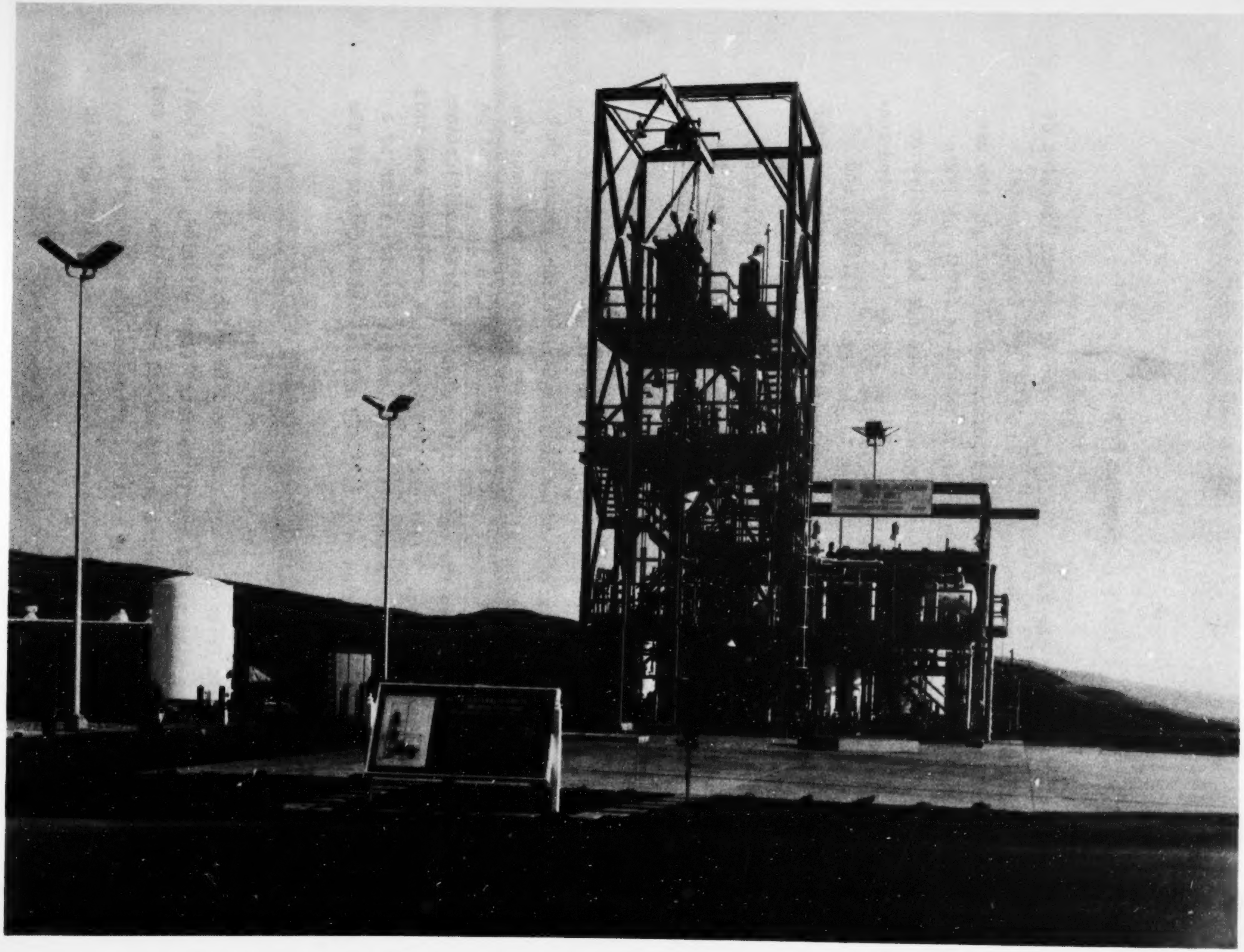
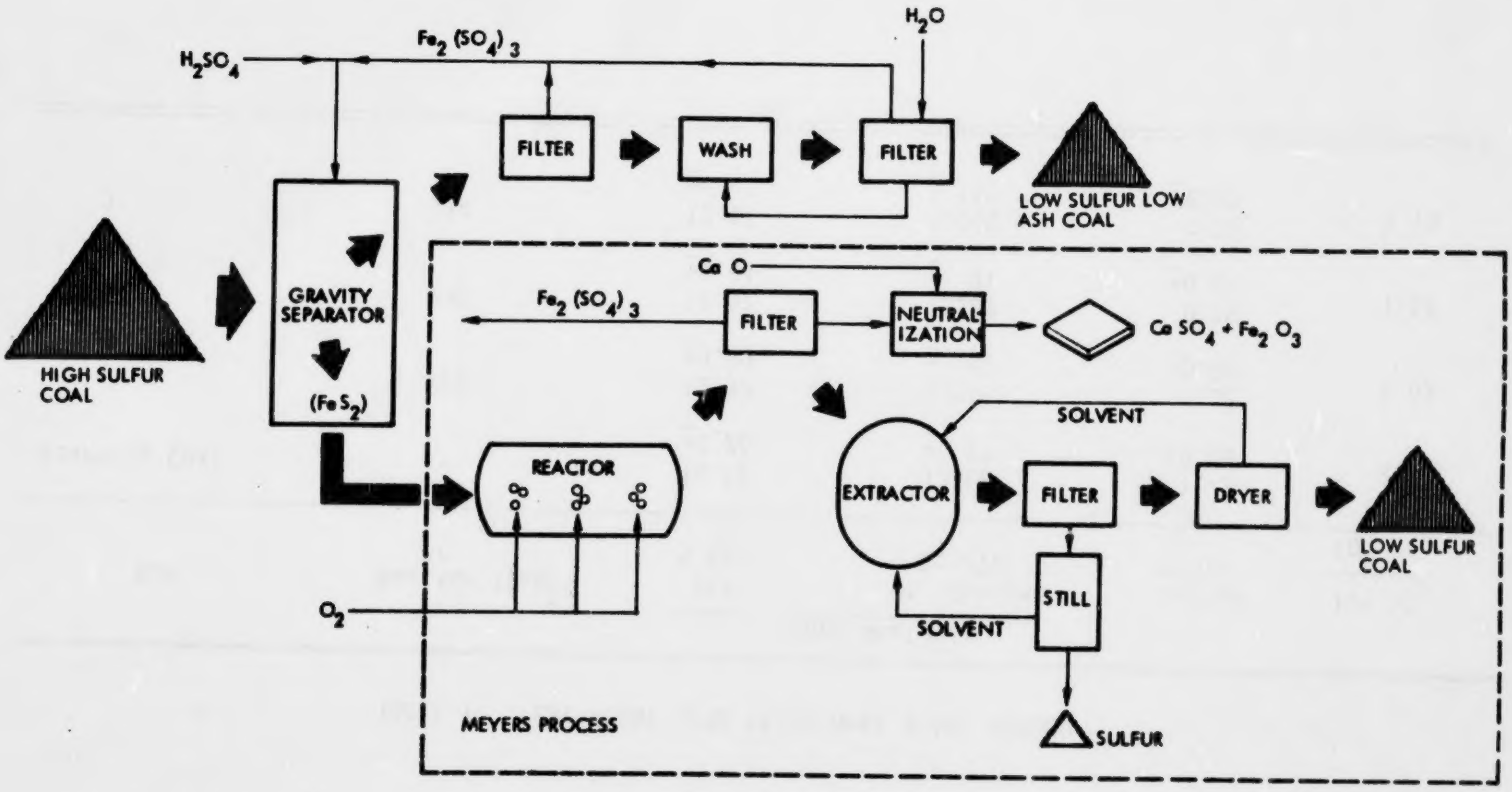


Figure 1. Federal Energy Technology Test Facility Sponsored by U.S. Environmental Protection Agency at TRW's San Juan Capistrano Test Site.

TABLE 1. TEST PLANT DATA TAKEN OVER 5-DAY PERIOD

Run	Reactor Temp., °F	Coal Analysis			
		Ash % w/w	Ht. Content Btu/lb	Sulfur % w/w	$\frac{\text{lbs } SO_2}{10^6 \text{ Btu}}$
Starting Coal	-	16.11 ±0.26	12508 ± 77	1.73 ±0.05	2.80
1	222	12.97 ±0.89	13258 ± 162	0.68 ±0.04	1.03
2	232	12.02 ±0.64	13388 ± 91	0.78 ±0.07	1.17
3	234	12.51 ±0.95	13265 ± 155	0.75 ±0.03	1.13



H_2SO_4
$Fe_2(SO_4)_3$
H_2O
FILTER
WASH
FILTER
LOW SULFUR LOW ASH COAL
HIGH SULFUR COAL
GRAVITY SEPARATOR
(FeS_2)
Ca O
$Fe_2(SO_4)_3$
FILTER
NEUTRALIZATION
$CaSO_4 + Fe_2O_3$
SOLVENT
REACTOR
EXTRACTOR
FILTER
DRYER
LOW SULFUR COAL
O_2
STILL
SOLVENT
MEYERS PROCESS
SULFUR

Figure 2. Gravichem Process.

Use of the leach solution gravity separation technique in conjunction with the Meyers Process results in an overall processing cost, including capital amortization, of $12/ton with 93-96% overall energy efficiency. This approach also led to a new and advanced method for physical cleaning of fine or gob coal.

This present contract was awarded with tasks for the experimental and applicability assessment of the Meyers Process.

The technical accomplishments of this contract modification for Preservation of the Coal Desulfurization RTU and Bench-Scale Testing are presented in the following sections:

Reactor Test Unit Inspection and Preservation;

Bench-Scale Extractions;

Specifications and Estimates for R-1 Reactor Replacement;

RTU Acetone Extraction Unit and

Application Studies

2.0 CONCLUSIONS

1. The inspection found the RTU to be in operating condition. However, corrosion during operation had created a need for early replacement of some equipment and components. The only condition of immediate concern is the severity of corrosion in reactor R-1 and associated pumps and plumbing. Corrosion observed in certain flanges and valve retainer seals (gasket sealing surfaces) and in the leach solution and coal slurry feed pumps are of only minor concern. These situations had been recognized earlier during RTU operation. Potential solutions have been identified, but further operation of the RTU will be needed to evaluate their effectiveness.

2. A replacement R-1 reactor can be built of titanium with excellent assurance of long-term serviceability.

3. Application of the Gravichem Process to Eastern and Midwestern steam-coal resources would reduce present oxides of sulfur emissions in the United States by 45-58%.

4. The Gravifloat portion of the Gravichem Process can recover depyrited, very low-ash and low-sulfur fuel from waste coal fines such as slurry ponds.

5. The Gravifloat coal in oil or water slurry is a potentially excellent fuel for use in switching oil-fired boilers to coal.

3.0 RECOMMENDATIONS

1. The Reactor Test Unit should be used for testing of a broad range of coal desulfurization processes after refurbishment or replacement of the R-1 reactor.

2. Further near-term coal desulfurization studies should concentrate on the removal of organic sulfur.

4.0 REACTOR TEST UNIT INSPECTION AND PRESERVATION

4.1 INTRODUCTION

During 1977 and early 1978, TRW supervised design and construction, and operated a Reactor Test Unit (RTU) designed to remove pyritic sulfur from coal. The purpose of these efforts was to demonstrate successful Meyers Process operability at pilot plant scale and to provide data for designing larger scale plants. In late January 1978, due to fiscal considerations, operation of the RTU was suspended. This work was sponsored by the Environmental Protection Agency (EPA) under Contract 68-02-1880.

In anticipation of restarting RTU operations in the future, EPA requested that TRW preserve and maintain the RTU in a standby condition. In addition to preparing and implementing a maintenance plan designed to minimize system deterioration, an inspection of equipment for evidence of corrosion and excessive wear was also to be conducted.

The purpose of this report is to document the maintenance plan as well as present the results of the indepth plant inspection. A brief description of the RTU and its operating history is provided as background. The actions taken to place the RTU in a standby mode are discussed as is the plan which has been implemented to minimize deterioration. The equipment inspected for corrosion and wear and the findings of the inspection are described.

4.2 BACKGROUND

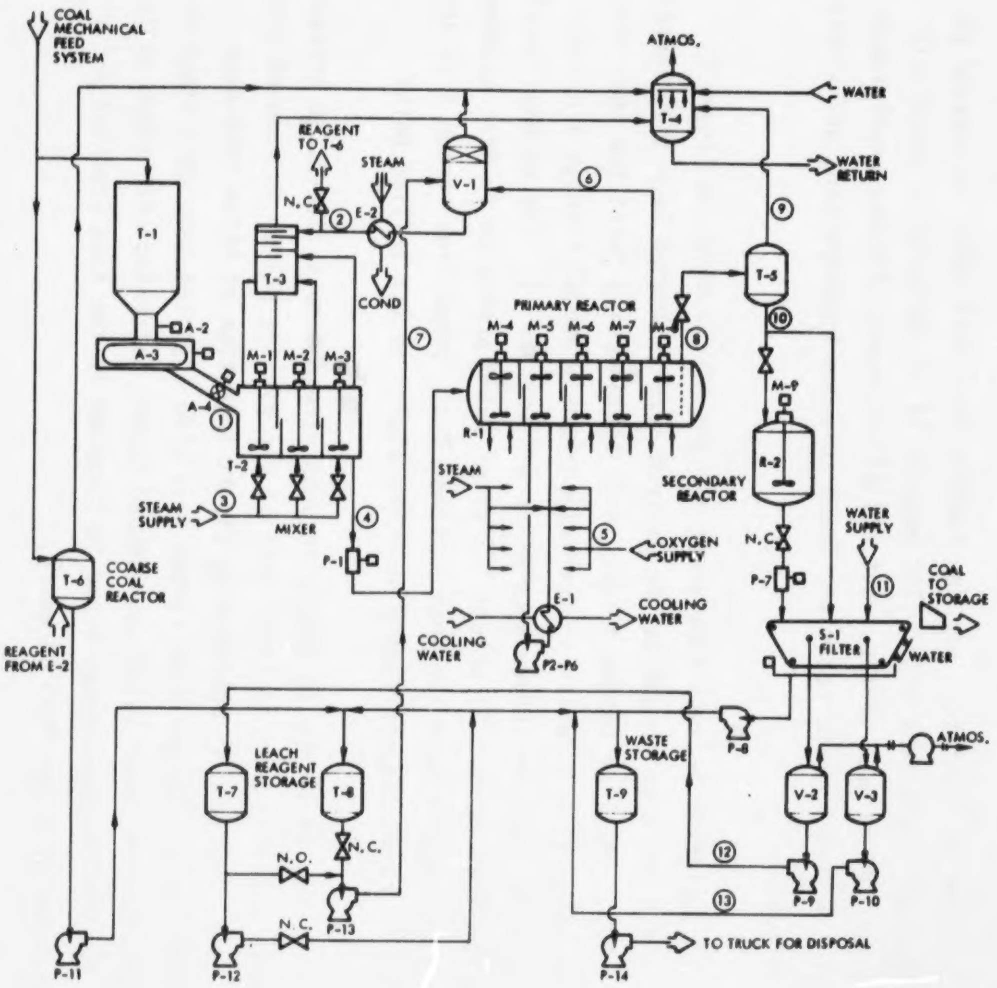
The Meyers Process is a chemical process for removing pyritic sulfur from coal. The coal is mixed with an aqueous solution of ferric sulfate. This slurry is then heated to 100-130°C where the ferric sulfate oxidizes the pyritic sulfur in the coal to form elemental sulfur and additional iron sulfate. At the same time oxygen is introduced into the aqueous solution to regenerate the spent ferric sulfate. The aqueous solution dissolves the iron sulfate and the elemental sulfur is removed by solvent extraction. The coal is dried and the solvent recovered. The by-products of the process are iron sulfate, which may be limed to give a dry gypsum and iron oxide material, and elemental sulfur. Both are safe, storable materials.

A three to eight tons per day Reactor Test Unit (RTU) is located at the TRW Capistrano Test Site. Its purpose is to demonstrate those unit operations which make up the front end of the Meyers Process: coal-leach solution mixing, pyrite reaction, leach solution regeneration, and slurry filtration.

A simplified process flow sheet for the RTU is shown in Figure 3. Coal which has been ground to the desired size is loaded into the storage tank, T-1. The live bottom feeder, A-2, continuously feeds the coal onto the weigh belt, A-3, which in turn discharges the coal through a rotary valve, A-4, into the three-stage mixer T-2 (stream 1). The aqueous leach solution feed (stream 2) enters T-2 after being preheated in heat exchanger E-2 and passed through the foam scrubber T-3. Steam from stream 3 is added to T-2 to raise the coal-leach solution slurry to its boiling point.

The heated slurry is pumped through stream 4 to a five-stage pressure vessel, reaction R-1, in which most of the pyritic sulfur is removed from the coal. Heating is achieved by direct injection of steam into each reactor stage. Oxygen from stream 5 is also injected into each stage to regenerate the leach solution. Unused oxygen saturated with steam exits the reactor through stream 6 and is scrubbed in the foam knockout V-1 with feed leach solution from stream 7.

The reacted coal slurry which is at elevated pressure and temperature, exits R-1 through stream 8 and is flashed into T-5. The steam generated in T-5 as well as vent gases from V-1 and T-3 are water scrubbed in T-4. The condensate and any entrained acid mist are removed with the return water.


COAL MECHANICAL FEED SYSTEM
ATMOS.
WATER
T-4
WATER RETURN
REAGENT TO T-6
STEAM
N.C.
E-2
V-1
T-1
T-3
COND
A-2
A-3
A-4
M-1
M-2
M-3
T-2
STEAM SUPPLY
MIXER
P-1
PRIMARY REACTOR
M-4
M-5
M-6
M-7
M-8
R-1
T-5
M-9
R-2
SECONDARY REACTOR
STEAM
OXYGEN SUPPLY
E-1
COOLING WATER
COOLING WATER
P2-P6
T-6
COARSE COAL REACTOR
REAGENT FROM E-2
N.C.
P-7
WATER SUPPLY
COAL TO STORAGE
S-1 FILTER
WATER
P-8
ATMOS.
LEACH REAGENT STORAGE
T-7
T-8
N.O.
N.C.
N.C.
P-11
P-12
P-13
WASTE STORAGE
T-9
V-2
V-3
P-9
P-10
P-14
TO TRUCK FOR DISPOSAL
①
②
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⑤
⑥
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⑬

Figure 3. RTU Schematic.

The reacted slurry is fed through stream 10 to the belt filter S-1. The filtrate (regenerated leach solution) removed from the coal is collected in the evacuated filtrate receiver V-2 and is pumped through stream 12 to one of two leach solution storage tanks, T-7 or T-8. Coal on the filter belt is washed with water from stream 11 and discharged to coal storage bins. The wash water is collected in the wash water receiver V-3 and pumped through stream 13 to the liquid waster tank T-9.

Upon completion of RTU shakedown, operational testing was initiated on 1 October 1977. During the next four months, the unit processed 49,700 pounds of coal over approximately 250 hours of operation. Sufficient data was acquired to verify the process chemistry and reaction rates, to parametrically evaluate process variables, to determine equipment operating characteristics, and to generate sufficient coal quantities for vendor testing. Operational testing was suspended on 26 January 1978.

4.3 ROUTINE MAINTENANCE DURING STANDBY

During early February 1978, the RTU coal desulfurization unit was secured for an extended shutdown period. The extent of the shutdown period was anticipated to be six to twelve months. TRW was requested to maintain the plant in a standby mode thus necessitating the continued lease of critical support equipment and the implementation of a minimum preventative maintenance program.

In order to secure the RTU and insure minimum damage to system components during the shutdown period, the following tasks were completed.

- Most RTU systems on-site were cleaned of residual acid and coal by flushing with water (some residual coal may still be in unaccessible lines). All tanks on the test stand were flushed and all pumps drained. Where equipment suppliers directed, preservatives (i.e., oils or inhibitors) were added to protect against corrosion.

- Electrical switches and breakers on decommissioned equipment were shut off, tagged and the main breakers thrown (off) and tagged. Guards on pumps were removed to permit periodic hand rotation (once a week) of the pump equipment.

- All instrumentation (i.e., Doric, Transducers, Flowmeters, and Level Devices) were sealed and desiccant added to keep moisture from contacting the equipment.

- The boiler was shut down and thoroughly dried to protect the rental equipment against corrosion.

- All liquid wastes (leach solution, dilute sulfuric acid, and waste water) were hauled away to the proper disposal areas by an outside contractor. The solid wastes (primarily processed coal) were hauled away to disposal also.

- Reactor R-1 was visually inspected and corrosion specimens removed for evaluation.

- Instrument air is being utilized to protect the Taylor Oxygen Analyzer AE-171 and the Autoweigh System A-3. No LN_2 supply has been maintained during shutdown. Residual feed coal in T-1 was removed and stored in two tote bins. The tanks in the tank farm were drained and manways opened.

- Twenty barrels of filter cake have been sealed, labeled, and stored in the RTU storage area.

A detailed listing of the specific shutdown related operations which were carried out is presented in Table 2. The table is divided into two groups, mechanical equipment related activities and those associated with instrumentation/electrical RTU components.

During the shutdown period, a minimum scheduled preventative maintenance program is being conducted. Those activities which are carried

TABLE 2. SPECIFIC RTU SHUTDOWN ACTIVIES

Mechanical Related

1. Cleaned out all piping, pumps, and valves in the RTU system and flushed with water to remove acid and coal.
2. Drained R-1 primary reactor and pumped slurry to tank T-9 in the tank farm.
3. Drained T-2 mixer of all slurry and pumped to tank farm.
4. Shut down all pumps and lubricated all parts of motors and bearings (P-1 through P-16).
5. Performed special cleaning of K-1 vacuum pump and added corrosion inhibitor.
6. P-15 pump flushed with clean water and isolated with feed valve open.
7. Air compressor serviced and lubricated.
8. Dcric shutdown for standby - batteries disconnected from unit.
9. Trailer (control center) leased for standby - roof retarred.
10. Boiler, LPC tanks (2), LN_2 tank leased for standby.
11. Forklift and liquid O_2 tank were returned to supplier and leases terminated.
12. Dumpsters (4) and waste bins (2) were returned to supplier and contract terminated.
13. Dumped coal from T-1 coal storage tank into tote bins.
14. Cleaned autoweigh system and purged with air.
15. Flushed all tanks and drained prior to applying N_2 blanket for standby.

(Continued)

TABLE 2. (Continued)

Mechanical Related (Continued)

16. Removed corrosion samples from R-1 primary reactor cell 3 and inline orifice specimens in recirculating loops.
17. Lubricated mixers.
18. Stencilled 20 coal product barrels with permanent labels.
19. Locked up all tools and special equipment in control center.
20. Inventoried all uninstalled parts, spares, etc.
21. Deactivated O_2 analyzer, purged with N_2 and isolated.
22. Cleaned V-1, V-2, and V-3.
23. Locked up all safety gear in the RTU storage shed.
24. Returned all cylinders for air breathing equipment.
25. Returned all cylinders of ultra pure O_2 to supplier.
26. Placed all bottled sulfuric acid in storage shed.
27. Cancelled all outstanding purchase orders.
28. Finished the fabrication of a Hasteloy C pump rotor and installed in P-1.

Instrumentation/Electrical Related

1. Control Console
    - Removed front panel of microprocessor and turned emergency battery power switch to off. Replaced front cover and turned off 115 VAC power.
    - Disconnected all 115 VAC power busses in control console from floor outlets. Coiled up all extension cords and connectors and stored in console.

(Continued)

TABLE 2. (Continued)

Instrumentation/Electrical Related (Continued)

2. Oxygen Analyzer

   - Plumbed instrument air to the inlet (downstream of remote solenoids) of the reference and sample cells.
   - Set reference cell to reference and sample cell to sample.
   - Set internal cabinet heat control for 70°F.
   - Tagged breaker 16 on LP-1 to read: "Do Not Turn Off Before Contacting W. Bowes/J. Hunt".

3. Autoweigh

   - Closed supply valve between autoweigh and T-1 tank.
   - Installed a blanking piece of metal between autoweigh discharge chute and T-2 inlet.
   - After autoweigh was cleaned and the cabinet resecured, plumbed instrument air into the existing N_2 purge point on the discharge duct.

4. Electrical 115 VAC Power

   - Turned off individual breakers located by the following instruments instruments: FE-31, FE-83 through FE-87, FE-29, FE-101, FE-157, and FE-158.
   - Turned off all breakers on LP-1 except 16.

5. Level, Steam, and Pressure Controllers

   - Closed all N_2 supply valves to LI-26, LI-58, LC-39, PIC-43, LI-95, LC-107, and LC-108.
   - Installed Humi Sorb desiccant bags in the following controllers and, utilizing masking tape, taped all door-to-cabinet seams: LT-26, TIC-32, LC-39, LI-58, PIC-43, LI-95, TIC-102, LC-107, LC-108, LI-130 through LI-132.

(Continued)

TABLE 2. (Continued)

Instrumentation/Electrical Related (Continued)

6. Motor Control Center, MCC-100
   - Turned off all breakers, except 1E, 6C, 7DL, 7AL, 7AR, and 9AR.
7. Emergency Lighting
   - Disconnected charger from 115 VAC source.
   - Disconnected emergency lights from battery.

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out are delineated in Table 3. As may be seen, the scheduled maintenace program has, as its primary objective, the preservation of all major rotating equipment. The tabulated maintenance program has been utilized since early February 1978.

4.4 RTU INSPECTION AND INSPECTION RESULTS

An inspection of the RTU conducted in December 1977 revealed that reactor R-1 and its associated piping had experienced crevice corrosion of varying degrees. However, leach solution feed lines, the slurry mix tank T-2, the flash-drum T-5, and reactor-regenerator pumps were unaffected. The reactor was repaired, the associated piping replaced and the RTU operation was continued. The extent of the December 1977 inspection was a cursory nature being limited by program pressures to continue process evaluations. The current lull in RTU operations has provided an opportunity to perform a more complete inspection.

Since complete disassembly and inspection of the entire RTU would be impractical, the 65 items listed in Table 4 were selected for inspection. The selection of these items was such that their observed condition would be an indication of the overall condition of the RTU. The selected items included major equipment, valves, piping, thermocouple probes, and flanges from various locations. They provide a basis for judging which areas within the RTU are prone to corrosion.

TABLE 3. RTU PREVENTIVE MAINTENANCE UTILIZED DURING SHUTDOWN

		Daily	Weekly	By Whom
1.	Trailer - Air conditioner check (70-75°C) operating	X		SOR
2.	Maintain pumps by rotating by hand - 180°C (to prevent flat spot on bearings)			
	P-2 Recir. pump		X	TRW M
	P-3 Recir. pump		X	TRW M
	P-4 Recir. pump		X	TRW M
	P-5 Recir. pump		X	TRW M
	P-6 Recir. pump		X	TRW M
	P-9 Filtrate pump		X	TRW M
	P-10 Wash pump		X	TRW M
	P-12 Leach solution pump		X	TRW M
	P-14 Waste disposal pump		X	TRW M
	P-15 Cooling water pump		X	TRW M
3.	K-1 Vacuum pump (rotate) by jogging pump electrically three times to prevent flat spots. (No liquid required.)		X	TRW M
4.	Air compressor			
	• Operate at pressure of 90-110 psi	X		TRW M
	• Drain air drier on compressor		X	TRW M
5.	LN_2 - Tank 103 LO_2 - Tank 102		NONE REQUIRED	

(Continued)

TABLE 3. (Continued)

	Daily	Weekly	By Whom
6. Cooling water pond			
• Water level		X	TRW M
• Water condition		X	TRW M
7. Oxygen analyzer			
• Cabinet heaters		X	TRW M
• Gas throughout		X	TRW M

Note:

SOR - TRW security personnel on their normal rounds.

TRW M - Assigned TRW Maintenance servicemen.

For the purpose of this report, the RTU has been divided into eight systems. Each system represents a different process function. Each has its own set of operating conditions in terms of temperature, pressure, and process fluid composition. The process conditions, the items inspected, and the inspection results for each process area are discussed in Sections 4.4.1 through 4.4.8.

To facilitate assembly and disassembly of equipment, gasketed joints were used throughout the RTU. Deterioration of some of the gasket materials and corrosion of the sealing surfaces were found during the inspection.

Since the anomalies appear to be related and because their occurrence appears to be independent of location within the RTU, they are discussed as a separate subject in Section 4.4.9.

During the course of the inspection activity, components removed and inspected were either left out of the RTU or reinstalled. Those items left out of the system have been tagged and stored in the equipment trailer. The associated connecting flanges were sealed with tape to preclude the

TABLE 4. RTU INSPECTION ITEMS

Number	Description
1	Hoist SP-2
2	Bin Tilt Mechanism
3	Lined Switch LSL-8 in Coal Storage Tank T-1
4	Coal Storage Tank T-1
5	Rubber Collar between Coal Storage Tank T-1 and Live Bottom Feeder A-2
6	Rubber Collar between Live Bottom Feeder A-2 and Weigh Belt A-3
7	Weigh Belt A-3
8	Rotary Valve A-4
9	Water Cooled Duct between Rotary Valve A-4 and Slurry Mix Tank
10	A Section of Process Line AA-1 between Slurry Mix Tank T-2 and Foam Scrubber
11	Leach Solution Flow Meter FE-31
12	A Section of Process Line between the Leach Solution Flow Meter FE-31 and the Foam Scrubber T-3
13	Level Transmitter LT-26 in Slurry Mix Tank T-2
14	Slurry Mix Tank T-2
15	Mixer M-3 in Cell 3 of Slurry Mix Tank T-2
16	Sample Valve M4-8 on Cell 3 of Slurry Mix Tank T-2
17	Thermocouple Probe TE-21 in Cell 3 of Slurry Mix Tank T-2
18	Slurry Feed Pump P-1
19	Slurry Flow Meter FE-101
20	A Section of Process Bins AA-7 Downstream of Slurry Flow Meter FE-101

(Continued)

TABLE 4. (Continued)

Number	Description
21	Valve MA-16 In-Process Bins Between Slurry Mix Tank T-2 and Waste Disposal Tank T-9
22	Valve MA-15 In-Process Line Between Slurry Mix Tank T-2 and Primary Reactor R-1
23	Leach Solution Heat Exchanger E-2
24	Slurry Feed Line AA-5 to Cell 3 of Reactor R-1
25	Slurry Feed Line AA-6 to Cell 1 of Reactor R-1
26	Knockout Drum V-1
27	Level Switch LSL-31 in Knockout Drum V-1
28	Level Controller LC-39 for Knockout Drum V-1
29	Oxygen Analyzer AE-171
30	Steam Feed Line AA-4 to Cell 1 of Reactor R-1
31	Cell 3 of Reactor R-1
32	Sample Valve on Cell 4 of Reactor R-1
33	Level Controller LT-48 on Cell 5 of Reactor R-1
34	Level Switch LSL-59 in Cell 5 of Reactor R-1
35	Thermocouple Probe TE-54 for Cell 3 of Reactor R-1
36	Thermocouple Probe TE-55 for Cell 4 of Reactor R-1
37	Thermocouple Probe TE-56 for Cell 5 of Reactor R-1
38	Low Level Valve LV-59 in Slurry Discharge Line from Cell 5 of Reactor R-1
39	Slurry Control Valve KV-241 in Discharge Line from Cell 5 of Reactor R-1
40	Gas Flow Meter FE-44
41	Thermocouple Probe TE-148 from Gas Effluent Line
42	Pressure Control Valve PV-43

(Continued)

TABLE 4. (Continued)

Number	Description
43	Flow Meter FE-83 in Recirculation Loop for Cell 1 of Reactor R-1
44	Flow Meter FE-84 in Recirculation Loop for Cell 2 of Reactor R-1
45	Flow Meter FE-85 in Recirculation Loop for Cell 3 of Reactor R-1
46	Flow Meter FE-86 in Recirculation Loop for Cell 4 of Reactor R-1
47	Flow Meter FE-87 in Recirculation Loop for Cell 5 of Reactor R-1
48	Recirculation Loop Plumbing for Cell 4 of Reactor R-1
49	Level Switch LSH-88 on Flash Drum T-5
50	Secondary Reactor R-2
51	Slurry Feed Pump P-7
52	Belt Filter S-1
53	Filter Belt Wash Pump P-8
54	Filtrate Pump P-9
55	Vacuum Pump K-1
56	Filtrate Line AA-8 between Filtrate Receiver V-2 and Filtrate Pump P-9
57	Leach Solution Storage Tank T-7
58	Leach Solution Storage Tank T-8
59	Liquid Waste Storage Tank T-9
60	Leach Solution Circulation Pump P-12
61	Leach Solution Feed Pump P-13
62	Basket Strainer SP-11

(Continued)

TABLE 4. (Continued)

Number	Description
63	Leach Solution Feed Line AA-3 Between Basket Strainer SP-11 and Leach Solution Feed Pump P-13
64	Valve T4-28 in Inlet Line to Liquid Waste Storage Tank T-9
65	Valve T4-6 in discharge line between Filter Cake Wash Pump P-10 and Liquid Waste Storage Tank T-9

introduction of foreign objects, insects, etc. No attempt was made to reseal flange joints upon reinstallation of components or equipment. Gaskets will have to be replaced and the flange joint sealed and leak checked. To assist prestart-up operations, the reinstalled joints have been tagged.

Figures 4 through 17 show the condition of the RTU after the disassembly inspection. The control console is shown in Figure 4 while a view of the west side of the RTU stand is shown in Figure 5. Views of the oxygen analyzer and bin tilter can be seen in Figures 6 and 7. The next two figures (Figures 8 and 9) show the equipment on level 4: the top of the coal feed tanks T-1 and T-6, the knockout drum V-1, the vent gas scrubber T-4, and their associated plumbing, valves and instrumentation.

Figure 10 is a view of the bottom of the coal feed tanks and the weigh belt feed A-3. General views of the mix tank T-2, the primary reactor R-1, and the belt filter S-1, are shown in Figures 11 through 14. Figures 15 and 16 show the filtrate collection equipment and the recirculation pumps for T-1, respectively. Figure 17 looks down on the north end of the tank farm. Tanks T-7 and T-8 and the mounting platforms for pumps P-12 and P-13 are shown. The various pictorial views of the RTU in essence show the current condition of the facility.

4.4.1 Coal Feed System

Feed coal is received from a subcontractor grinding facility in steel bins. These bins are hoisted to the top of the RTU test stand and emptied by a pneumatic bin tilter A-1. The coal is routed through a duct to the coal storage tank T-1. A vibrating, live bottom feeder, A-2, at the bottom of T-1 prevents solids bridging and maintains coal flow to the weigh belt A-3. The coal is discharged from A-3 through a rotary valve into a water cooled duct which leads to the coal-leach solution mixer T-2.

During operation, the bin tilter, storage tank, weigh belt, and ducting are maintained at a positive pressure with nitrogen (N_2) to preclude oxygen or moisture contaminating the feed coal. The coal bins are also pressurized with N_2 at the time of the filling for the same reason. No attempt is made to heat or cool any of this equipment; it operates at ambient temperature.

During RTU operations, two coal bins would not mate properly with the hoist attach mechanisms. The attaching interface structure at the top of these bins appears to have been bent. Consequently, these bins cannot be lifted by the hoist. Visual inspection indicates that the remaining 73 bins are serviceable.

The electrically operated hoist undergoes semi-annual inspection and load tests as required by the safety code. During the last inspection, the hoist was found to be inoperable. There is an open circuit in the hoist control circuit.

The bin tilter A-1 is equipped with a T-handle wrench for opening the bin discharge port after the coal bin has been placed on the bin tilter. The cloth boot sealing the wrench to the tilter is worn and may leak coal dust during coal discharge operations. Otherwise the bin tilter appears to be in good condition.

The eight-inch diameter at the top of T-1 was opened and the interior of the coal feed tank inspected. The low level alarm switch LSL-8 was also removed. No visual anomalies were observed.

The rubber collar connecting the live bottom feeder A-2 to the coal feed tank is cracked approximately 50% around its circumference (see Figure 18). However, the rubber collapse between the live bottom feeder and the weigh belt A-3 is not damaged.

Visual inspection of the weigh belt mechanism inside its cabinet revealed only slight rust on the belt adjustment bolts. However, the seals for the access door cover and windows have deteriorated and will probably need replacing prior to reinitiation of plant operations.

The rotary valve A-4 was removed from the drive unit and the bearings were checked for both side and end play. None was found. The clearances between the rotor vanes and the valve body are not badly worn. However, one of the vanes is bent (see Figure 19). The damage was undoubtedly caused by some foreign object being caught between the rotor and the valve body while the valve was in operation.

The water cooled duct between the rotary valve A-4 and the slurry mix tank T-2 was found to be in good condition with no visible corrosion present.

4.4.2 Leach Solution and Waste Liquid Storage System

This system consists of the three large storage tanks T-7, T-8, and T-9 as well as the leach solution circulation pump P-12 and the piping, valves, etc., generally located in the tank farm. The process environments experienced by this equipment are ambient temperature and pressure.

The manways on tanks T-7, T-8 and T-9 were opened and the tank interiors inspected. The manway seals and tank internal components are in good shape. The floors of T-7 and T-8 are coated with a reddish brown residue approximately 1/4-inch thick. Since these tanks contained either fresh or recovered leach solution, then residue is probably residual iron sulfate left after the tanks were drained.

Tank T-9 was used to collect the coal wash liquid from the S-1 filter and the material flushed from the RTU during shutdown operations. The bottom of the tank is coated with coal fines and what appears to be elemental sulfur or yellow-boy sulfate deposited from evaporated residual liquid. The internal walls are also coated to a height of approximately 15 feet with what appears to be an opaque substance which resembles tar.

When the drain plug was removed from the bottom of the leach solution circulation pump P-12, approximately one cup of liquid was removed. Subsequent disassembly inspection revealed minor surface etching on the lower one-fourth of the impeller housing and impeller back plate (see Figure 20). The impeller, the shaft, and mechanical shaft seal are in good shape.

Since the pump backing plate and impeller housing were etched only on the lower 1/4 of the surface, it is believed that the residual liquid was the cause.

The isolation valve T4-28 on the inner line to T-9 as well as a Jamesbury valve T4-6 in the line from the wash water receiver V-3 to T-9 were disassembled and inspected. Some coal and salt residue was found in the T4-28 valve cavity. Slight crevice corrosion was also found on some of the gasket seal surfaces. This type of observations is discussed in detail in Section 4.4.9 of this report. No other defects were found.

4.4.3 Leach Solution Feed System

During the RTU operation, pump P-13 is used to pump leach solution from T-7 or T-8 through a strainer S-11 to the knockout drum V-1 where the leach solution is used to break down any foam and demist the vent gases from reactor R-1. The leach solution is then fed through heat exchanger E-2 for preheating to 170°C and then into the foam scrubber T-3. The leach solution is used in T-3 to break down any foam generated during the mixing of leach solution with the coal in T-2. The leach solution is gravity fed from T-3 to the first stage of the T-2 mixer. Leach solution feed rates are monitored by a magnetic flow meter FE-31 located betweeen the heat exchanger E-2 and the foam scrubber T-3.

The leach solution leaving the storage tank may or may not contain any dissolved oxygen depending upon whether the solution has been used in a previous run or is a fresh batch. In either case the solution is assumed to become nearly saturated at ambient temperature with oxygen in V-1. Solution temperature is subsequently raised to approximately 170°F in the heat exchanger and remains near that temperature until it reaches the mix tank T-2.

The items inspected included strainer SP-11, the process line AA-3 between the strainer and the pump P-13, the knockout drum V-1, the solution level controller, LC-39 and the low level alarm sensor LSL-37 on V-1, the heat exchanger E-2, the flow meter FE-31, a short section of process line AA-2 immediately down stream of the flow meter, and a section of the leach solution discharge line AA-1 from T-3.

The basket strainer SP-11 and the process line AA-3 from the strainer to pump P-13 appears to be in very good condition. No evidence of corrosion (except that reported in Section 4.4.9) or other anomalies were found.

As noted in the final report, EPA-600/7-79-013a, "Reactor Test Project for Chemical Removal of Pyritic Sulfur from Coal," dated January 1979, pump P-13 experienced a rotor related problem near the end of RTU operation. Disassembly inspection at that time revealed that the chrome plate on the rotor had crazed and flaked off (see Figure 21) increasing the clearance between the rotor and pump stator. This excess clearance permitted back flow of the leach solution and thereby limited the pumping capability. The P-13 rotor was replated with chrome and placed back into service. A new rotor was also fabricated from 316L stainless steel (unplated) for use as a spare.

The most recent inspection shows the pump stator and replated rotor to be in a good condition. There is no evidence of corrosion, and a "tight" fit still exists between rotor and stator. This tight fit indicates that the stator has not worn appreciably. The outside diameter of the rotor (lobe-to-lobe) measures 1.875 inches. This is within the tolerances for a new rotor. However, the surfaces of the shaft pins which are normally exposed to the leach solution are corroded. The condition of these pins are shown in Figure 22.

The knockout drum V-1 as well as the solution level controller LC-39 and low level switch LSL-37 are also in very good condition. However, the bubble cap trays are jammed in place and could not be removed without first removing V-1 from the RTU structure. Because the internal surfaces visible from the top of V-1 are in excellent condition with no signs of corrosion (see Figure 23), it was decided not to remove V-1 from the RTU stand for further disassembly.

The internal surfaces of heat exchanger E-2, flow meter FE-31 and the process pipes AA-2 and AA-1 are in excellent shape. No evidence of corrosion or other anomalies were found.

4.4.4 Coal Slurry Preparation System

The equipment in this portion of the RTU consists of the slurry mix tank T-2, a slurry feed pump P-1, a flow meter FE-101, and the piping which connects these pieces of equipment and subsequently leads to reactor R-1.

The mix tank T-2 contains three cells each equipped with a propeller blade mixer, steam injection port, and a thermocouple well. The cells are separated by overflow wiers which can be raised and lowered to control the volume of slurry contained the first two cells.

Both leach solution at 170°C and dry coal at ambient temperature are continuously fed into cell 1. The slurry is heated by direct steam injection into each cell. The temperature in the three cells is typically 180°F, 205°F, and 215°F (cells 1 through 3, respectively). The heated slurry is discharged from cell 3 through the slurry pump P-1 to either cell 1 or cell 3 of reactor R-1. A level control sensor LT-26 controls the speed of P-1 so that the volume of slurry in cell 3 of T-2 is held at a predetermined level. The flow meter is located between P-1 and R-1.

The pressure in the system is atmospheric until the solution reaches P-1 where it is increased to the operating pressure of the reactor R-1, typically 50 to 100 psig.

The disassembly inspection of the slurry mix tank T-2 consisted of opening the manway into cell 2 and removing the thermowell TE-21, the sample valve M4-8 and the level controller LT-26 from cell 3. This hardware is in very good condition. The mixer shaft and blades as well as the baffles between the cells do not have any evidence of corrosion (see Figures 24 and 25). However, the agitator blade shows evidence of slight mechanical abrasion as shown in Figure 26. The portion of the level controller that extends into the mix tank shows no evidence of corrosion. There has been some mechanical abrasion of the tube, but only to the point of brightening the surface. There are two small corrosion pits near the end of the thermowell probe on TE-21 removed from cell 3. These are in the weld material rather than in the parent material (see Figure 27). There also are small corrosion pits in the weld material connecting the thermocouple well flange to the T-2 tank wall. These are shown in Figure 28. The sample valve M4-8 taken from cell 3 was also disassembled and visually inspected. No corrosion or other anomalies were found.

The results of the disassembly inspection of both slurry feed pumps are reported here. The two pumps are identical and have seen approximately the same length of service functioning as P-1, i.e., pumping hot coal slurry from mix tank T-2 to reactor R-1. The flow sheet in Figure 3 shows P-7 as the discharge pump for reactor R-2. However, P-7 was used very little in this capacity.

During RTU operation, both pumps experienced the same difficulties experienced by the leach solution feed pump P-13, i.e., erosion and corrosion of the chrome plate on the rotors. However, in the case of the slurry pumps, the rotor SS-316L base material eroded and corroded causing loss of pumping capability. The cause of this additional erosion/corrosion is believed to be the abrasive nature of the coal slurry as opposed to the liquid leach solution handled by P-13.

The rotors from both P-1 and P-7 were replated to the required tolerances with chrome plate. P-1 was put back into service and performed satisfactorily for several hours before the chrome plate flaked off causing pump failure. Figure 29 shows the flaked condition. The thicker chrome plate could not withstand the thermal expansion from ambient to operating temperatures. Near the end of RTU operation, a new rotor was fabricated of Hastelloy C-276 and installed in P-1. P-7 equipped with a replated rotor was in service at the end of RTU operation

As expected, P-1 is in very good condition. The only sign of excessive wear is on a retaining ring for the pin at the small end of the rotor shaft. Measurements show that the diametrical clearance between the shaft and the retaining ring is .011 inches. It should be .001 inches.

Also as expected, the internal parts of pump P-7 show evidence of corrosion and wear. Figures 30 and 31 show the wear and corrosion at the drive end and the center section of the rotor. The intermediate shaft is etched in the area of the lip seal (see Figure 32). The universal linkage shaft is severely etched (see Figure 33). The shaft pins and pin retaining ring are also corroded (Figures 34 and 35, respectively).

The weld material conncecting the flange, which mates the P-1 discharge port with the downstream process line, is corroded (Figure 36). The corrosion is similar in type and extent to that found in the weld material connecting the thermocouple well flange to the T-2 vessel wall.

The flow meter FE-101, which measures the slurry flow rate from the mix tank T-2 to the reactor R-1, and the process line immediately downstream of the flow meter were removed, disassembled, and inspected. The process line included two ball valves, M4-15 and M4-16. The teflon line inside the flow meter had extruded slightly at the flanges. The flow straightness insert at the inlet to the flow meter had some corrosion pits. Neither condition has progressed enough to be of any concern. Except for very minor crevice corrosion on gasket seal surfaces (discussed in Section 4.4.9 of this report), the process line and ball valves are in excellent condition. This is also true for slurry feed lines and valves leading to cell 1 and cell 3 of reactor R-1 which were removed and inspected.

4.4.5 Primary Reactor System

The primary coal reactor R-1 is a horizontal pressure vessel 38 inches in diameter and 14.75 feet long. It is divided into five cells by stationary weirs which are 28.5 inches high. Each cell is equipped with a variable speed agitator and a slurry recirculation system. Hot slurry may be fed into either cell 1 or cell 3 and overflows into each succeeding cell unit until it reaches cell 5. The reacted slurry is discharged from cell 5 through a discharge valve KV-241 into the flash drum T-5. A buoyant type level gauge is used to monitor the slurry level in cell 5.

The slurry recirculation system for each reactor cell withdraws slurry from the cell, into a process line, through a centrifugal pump, and then returns to the same reactor cell. Reactor heating and slurry oxygenation are performed by injecting steam and oxygen into each slurry recirculation loop. Excess gases vent through a vent line leading from the top of the reactor to the knockout drum V-1. Reactor pressure is maintained by a pressure control valve and is monitored by a pressure transducer located in the gas vent system described later.

Each reactor cell is equipped with a thermocouple probe for monitoring slurry temperatures. Each cell and each recirculation loop also have a one-inch sampling port for withdrawing slurry samples for chemical analyses. The reactor R-1 and associated equipment is subjected to temperatures up to 275°F and to oxygen dissolved in the slurry.

As reported in the final report cited earlier, the reactor vessel, the reactor internals, and the slurry recirculation loop equipment experienced progressively severe corrosion, both crevice corrosion and pitting. The severity of the corrosion led to the recommendation to replace the reactor system with new reactor, piping, flanges, and valves constructed of titanium. The recommended material of construction is predicted not only by the results from materials study (also reported in the final report) but also by the size of the equipment. The RTU reactor and piping are too small for metallic, elastomer, and/or acid brick lining to be applicable. The recommendation is also based upon the relative low cost of titanium replacement parts. For example, a quote of $49,300 was obtained in January 1978 for a replacement titanium reactor vessel. The original non-titanium (stainless steel) reactor cost $25,900 in early 1976.

Figure 37 shows examples of the corrosion in cell 3 of the reactor. The severity is progressively worse in cells 4 and 4. Figures 38 through 40 show examples of pitting corrosion found in piping, valves, and on thermocouple probes. It should be noticed that these items were installed new in December 1977.

The centrifugal pumps in the slurry recirculation loops had not been disassembled during the previous inspections of the reactor system. Therefore, pump P-5 (servicing cell 4) was subjected to a disassembly inspection this time. The pump parts which had been exposed to the slurry show evidence of significant corrosion. Some of the corrosion can be seen in Figures 41 through 48. The corrosion on the pump impeller shown in Figure 45 appears to be associated with casting flaws. Figure 48 shows the eroded and etched surface found on the pump shaft.

The low level alarm switch LSL-59 and the level monitor gauge LT-58 were removed from cell 5 of the reactor and inspected. The only corrosion found was some pits on the outside diameter of the float well shown in Figure 49. The flow meter FE-86 in the recirculation loop for cell 4 was also found to be in excellent shape. The teflon liner was not deformed. There were no signs of leakage through the liner.

4.4.6 Gas Vent System

The vapor spaces of each of the three cells of the slurry mix tank T-2 are vented to the foam scrubber T-3. The vent gases are passed through a bubble cap tray flooded with hot leach solution to break up any foam which might be generated in the mix tank. Gases from the foam scrubber T-3 (predominately nitrogen from purge lines in the coal feed system) are vented to the vent gas scrubber T-4. The line from cell 1 of the mix tank T-2 and the foam scrubber T-3 was removed and inspected. No evidence of corrosion was found.

The vapor space in the primary reactor R-1 is vented to the knockout drum V-1 where the vent gases are contacted with unheated leach solution. This removes any steam or acid mist that might be entrained. The gases, predominently oxygen, exiting the knockout drum V-1, are vented to the vent gas scrubber T-4. The instruments that monitor and control the pressure in the knockout drum V-1 and the primary reactor T-1 as well as monitor the flow rate, temperature, and oxygen content of the vent gas from the reactor R-1 are located in the vent line between the knockdown scrubber V-1 and the vent gas scrubber. The flow meter FE-44, the thermocouple probe TE-148, and the pressure control valve PV-43 were removed and inspected. No anomalies were found. Some traces of particulate contamination (coal fines and evaporation residue) were noticed.

The oxygen analyzer AE-171 also appears to be in excellent condition. The interior of the cabinet is clean and no deterioration of flow lines and instruments could be detected.

All of the vent gases from the RTU pass through the vent gas scrubber T-4. It is here that the gases are washed with water from a spray head to remove any steam vapors or acid mist which might travel this far. Because this scrubber is constructed of fiber reinforced plastic and because the operating condition is benign, no inspection was made of this piece of equipment.

4.4.7 Secondary Reactor System

The secondary coal reactor R-2 is a 365 gallon, vertical tank equipped with a variable speed agitator, a level sensor LT-95, and a thermocouple probe TE-99. The original purpose of reactor R-2 was to enable us to further process coal slurries reacted in reactor R-1. Since the reactor rates experienced in R-1 were much higher than expected, R-2 was never used as a secondary slurry reactor. It was only used to prepare feed leach solutions. The leach solutions were prepared by sequentially adding under agitation sulfuric acid and ferric sulfate to distilled water previously charged to R-2. After the ferric sulfate dissolved, the leach solution was transferred to a leach solution storage tank T-7 or T-8.

Inspection of the secondary reactor R-2 consisted of opening the 18-inch diameter manway and visually inspecting the inside surfaces. It is in excellent condition. A view inside R-2 through the manway is shown in Figure 50.

4.4.8 Filtration and Leach Solution Recovery System

Processed slurries are discharged from the pressurized reactor R-1 into the flash drum T-5 which operates at atmospheric pressure. A porous demister pad is situated immediately above the slurry inlet. Even so, the flashed steam and gases from T-5 are vented through the vent gas scrubber T-4. The underflow slurry is gravity fed from T-5 to the filter S-1 for leach solution recovery and coal washing. The slurry level in T-5 is monitored by a level switch LSH-88 which triggers an alarm if the level becomes too high.

S-1 is a belt filter equipped with two filtration zones each having a discrete filtrate collector system. The filter belt consists of a polypropylene mesh cloth outer belt supported by a channeled rubber inner belt. Feed slurry, typically at 213°F, from T-5 is distributed on the belt by a stationary spreader. The leach solution is removed from the coal in the first filtration zone, collected in a filtrate receiver V-2, and recycled through pump P-9 to either storage tank T-7 or T-8. After this initial filtration, the filter cake is steamed and sprayed with hot water from overhead nozzles. The filtrate from cake wash is removed from filter cake in the second filtration zone. This filtrate is collected in the wash water receiver V-3 and transferred by pump P-10 to the waste disposal tank T-9. The filter cake is discharged from S-1 through a chute, a water spray is used to remove remaining coal particles from the outer belt. This prevents blinding of the belt. The belt wash is collected in a tray under the filter which drains to the filter wash pump T-8. The pump discharges the belt wash effluent to either the cake wash section of the filter or to the waste disposal tank T-9. The filtration unit is completely enclosed in a reinforced fiberglass hood. Sampling ports built into the hood permit sampling processed coal before and after the wash operation.

Inspection of the flash drum T-5 consisted of removal of the level switch LSH-88 from the bottom of the tank. The switch probe and the internal parts of T-5 visible through the open port are in very good condition.

During RTU operation, the inner rubber belt failed. The belt had softened due to prolonged exposure to the hot leach solution and began to adhere to the lip of the vacuum pans. The belt was literally pulled apart. The belt was repaired, and the vacuum pan edges were fitted with teflon overlays to preclude adherence at the belt-vacuum pan interfaces. The filter operated properly for the duration of operation.

The latest inspection shows that the inner and outer belts are intact and have not deteriorated since RTU shutdown. The cloth outer belt is worn from use and should be replaced when RTU operation is resumed.

All of the fiberglass components show no effects of weathering. However, some of the rubber seals and the belt dams are beginning to crack. Some of the metal shafts are also beginning to rust slightly. Electrical power to the belt drive was turned on. No problems were encountered.

The filtrate pump P-9 was removed and disassembled for inspection. A light reddish brown residue was found on internal surfaces. Wire brushing these surfaces did not reveal any evidence of corrosion. The pump seals are slightly worn, but are otherwise in good condition.

The same reddish brown residue was also found in the process line AA-8 leading from the filtrate receiver V-2 to the filtrate pump P-9. The pipe and flanges are in good condition; no sign of any corrosion.

Since RTU shutdown, the vacuum pump K-1 has been turned on regularly to prevent the seals from freezing to the shafts. Conversations with the manufacturer resulted in their recommendation not to disassemble the pump. They feel that the operating history is too short to justify any disassembly at this time. Their recommendation was honored.

The belt ash pump P-8 was disassembled and inspected. No corrosion or abrasion was found.

4.4.9 Gasket Sealing Surfaces

The defect found most frequently during the inspection was crevice corrosion of gasket sealing surfaces such as flange faces and valve seal retainers. Even so, only 30% of those surfaces inspected showed any evidence of corrosion. The severity of most of those was very slight. It was only those sealing surfaces exposed to high processing temperatures and high oxygen concentrations that were corroded severely enough to be of concern.

Apparently an electrolytic corrosion cell is initiated by leach solution seeping under the gasket material. The driving force is probably the oxygen concentration gradient between the solution under the gasket (low concentration) and the bulk of the solution in the immediate vicinity (high concentration). With this mechanism, the severity of the corrosion would be expected to increase with increased dissolved oxygen concentration and with increased temperature. This was indeed the pattern observed during the inspection. However, the temperature seems to have a greater effect than the dissolved oxygen concentration.

Figures 51 through 58 show the very slight crevice corrosion found on flange faces in process lines containing cold leach solution. Likewise, Figure 59 shows the very slight crevice corrosion found on the seal retainers in valve T4-6 taken from the filter cake wash line from filter S-1 to waste tank T-9. The concentration of dissolved oxygen in the leach solution located in these process lines is very low.

Figures 60 through 62 show the very slight crevice corrosion found on flange faces from the foam knockdown drum V-1. The leach solution in V-1 is at room temperature, but is also nearly saturated with oxygen from the vent gas from reactor R-1. Figures 63 and 64 also show the presence of very slight crevice corrosion on mating flanges at the V-1 gas discharge port. This represents the only incidence of corrosion found in vapor space portions of the RTU. However, leach solution probably reached this area when V-1 was inadvertently flooded during one of the early experimental runs. The flange had not been disassembled prior to this inspection.

Figures 65 and 66 show the crevice corrosion evident on mating flanges at the outlet of the E-2 heat exchanger. The leach solution from V-1 is heated to approximately 170°F by this heat exchanger. Again, the severity is very slight.

It is in T-2 that the effect of temperature on the crevice corrosion can be seen. The temperature in the first of the three cells in T-2 is typically 180°F. The temperature in cells 2 and 3 is 205°F and 215°F, respectively. Figure 67 is a close-up picture of the crevice corrosion found on the manway cover, which is on cell 2. The corrosion is still slight, but noticeably deeper than that observed in cooler sections. Figures 68 and 69 show slightly more extensive corrosion found on the mating flanges for the cell 3 thermocouple probe. Similar degree of crevice corrosion on the outlet flanges of pumps P-1 and P-7 are shown in Figures 70 and 71. Both of these pumps have been used to transfer hot slurry from the mix tank T-2 to reactor R-1. Figure 72 shows the slight crevice corrosion on a flange face taken from the slurry feed line leading from pump P-1 to cell 3 of the reactor R-1.

The most extreme operating conditions anywhere in the RTU are in reactor R-1. The slurry is at the highest temperature 230°F to 275°F, and saturated with oxygen at the operating pressure, 50 to 100 psi. As a result, the crevice corrosion is the most severe in the reactory system. An extreme example of crevice corrosion experienced under these conditions is shown in Figure 73. This picture shows the inside of the manway cover located on cell 3 of reactor R-1. The manway cover had been used to test an elastomer material as a potential tank liner. During RTU operation, the adhesive failed between the upper portion of the liner and the manway cover creating a crevice between the two. Figures 74 through 79 show the moderate crevice corrosion found on flange faces at the R-1 discharge valve assembly. This assembly was comprised of two remote actuated bell valves. One of these valves, LV-59, was normally open during RTU operation and only closed when the low level sensor in R-1 indicated that the slurry level in cell 5 was too low. The second valve, KV-241, was opened or closed by a timer to meter the flow of slurry out of the reactor.

Figures 80 through 83 show the crevice corrosion found on the flange faces and valve seal retainers located in other parts of the reactor system. The degree of corrosion is less than that experienced on the discharge valve assembly. However, the discharge valve assembly was installed during RTU construction. The equipment shown in the figures were installed in December 1977.

Except for those sealing surfaces located in the reactor system, the observed crevice corrosion does not present a concern regarding the near term operation of the RTU. Operation could continue for some time before leach solution leakage would occur. Even then, the defect would probably manifest itself as a slow seepage of leach solution.

However, the location and frequency of the observed crevice corrosion suggests that seepage of leach solution under the gasket must occur to initiate the defect. The obvious preventative is to utilize a better gasket seal. Two types of gaskets have been used to date: thin garlock and thicker polymeric material (EPDM). Neither is completely effective. It is recommended that a non-curing adhesive type material be tried between the gasket and the metal sealing surface. Both surfaces can be completely wetted with this adhesive thus preventing the initial seepage of leach solution into the interface. The EPDM gaskets also developed circumferential cracks inside and under the inside diameter of the sealing surface. Examples are shown in Figures 84 through 87. The pattern of the cracks suggest that the EPDM has deteriorated and that the flange bolts may have been over-tightened. The only other gasket found cracked is shown in Figure 88. This gasket is from the flange joint on the discharge part of the filtrate receiver V-2. It is cloth reinforced polymeric material. Since this gasket was installed by the manufacturers of the filtrate collection equipment, efforts are underway to identify the gasket material.

4.4.10 Equipment Spare Parts

As part of the RTU inspection, spare parts for the major pieces of equipment were inventoried. In addition to numerous valve and mechanical seal kits, the inventory consists of:

1 Cloth filter belt, P/N GB3725KVK, for S-1 filter
1 Inner belt for S-1 filter
1 Leach solution feed pump P-13 consisting of one each:
  - Moyano pump frame 3MA, Form FA, S/N AS-67166
  - Carter vari-speed reduction gear, Series 1DNRS
  - Reliance motor, P/N P14G2402S-XC, 1-1/2 HP
  - Foxboro speed control, Type C, S/N 3386296

1 Duplex strainer (SP-11), 2", stainless steel
2 Fischer-Porter magnetic flow meters, Mod 10D1418A, 50 GPM max
2 Signal converters, PR-50, for magnetic flow meters
1 Pump rotor for P-1, 316SS
1 Billet for pump rotor, Hastelloy C
2 ITT Barton flow meters, turbine type

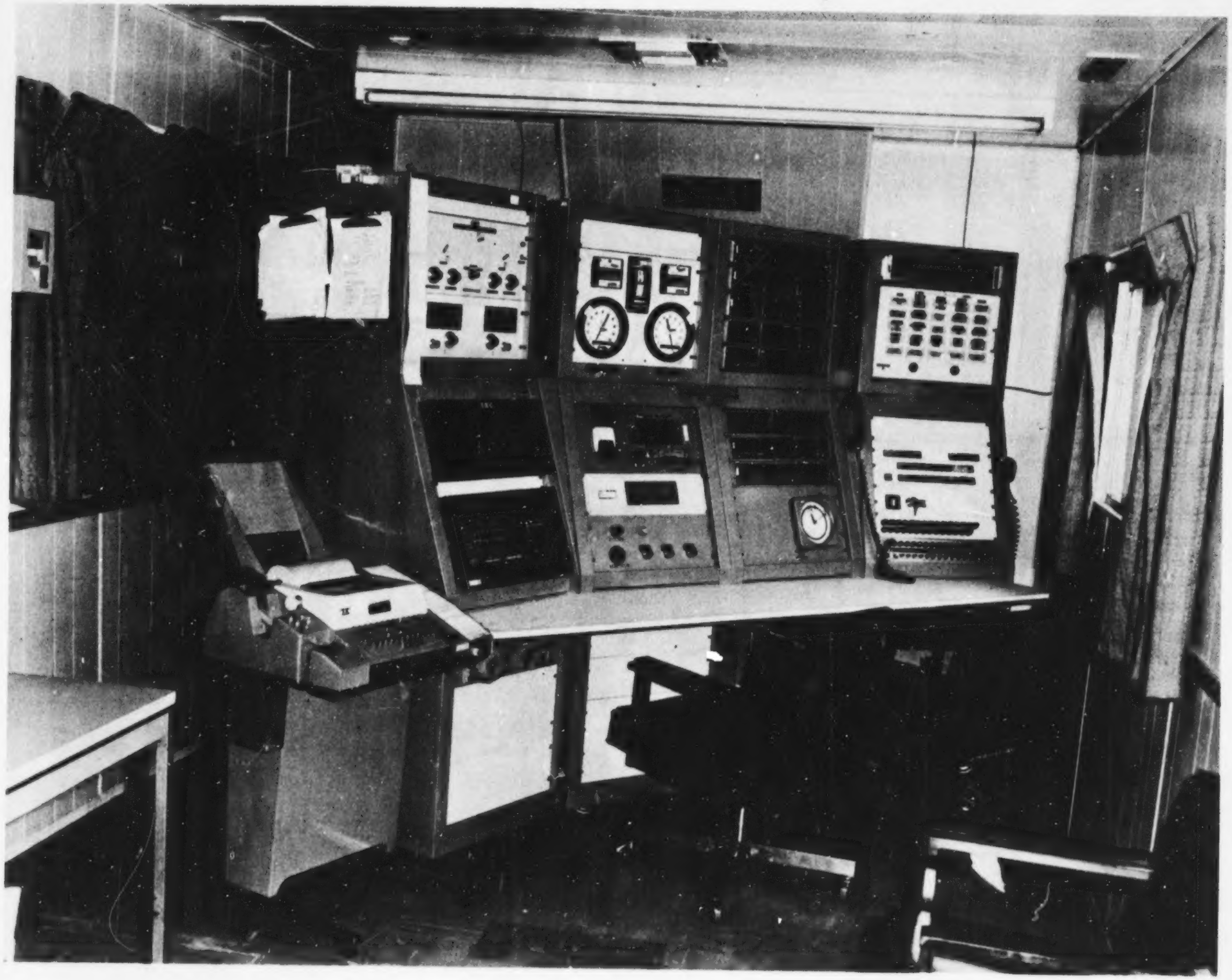


Figure 4. RTU Control Console.

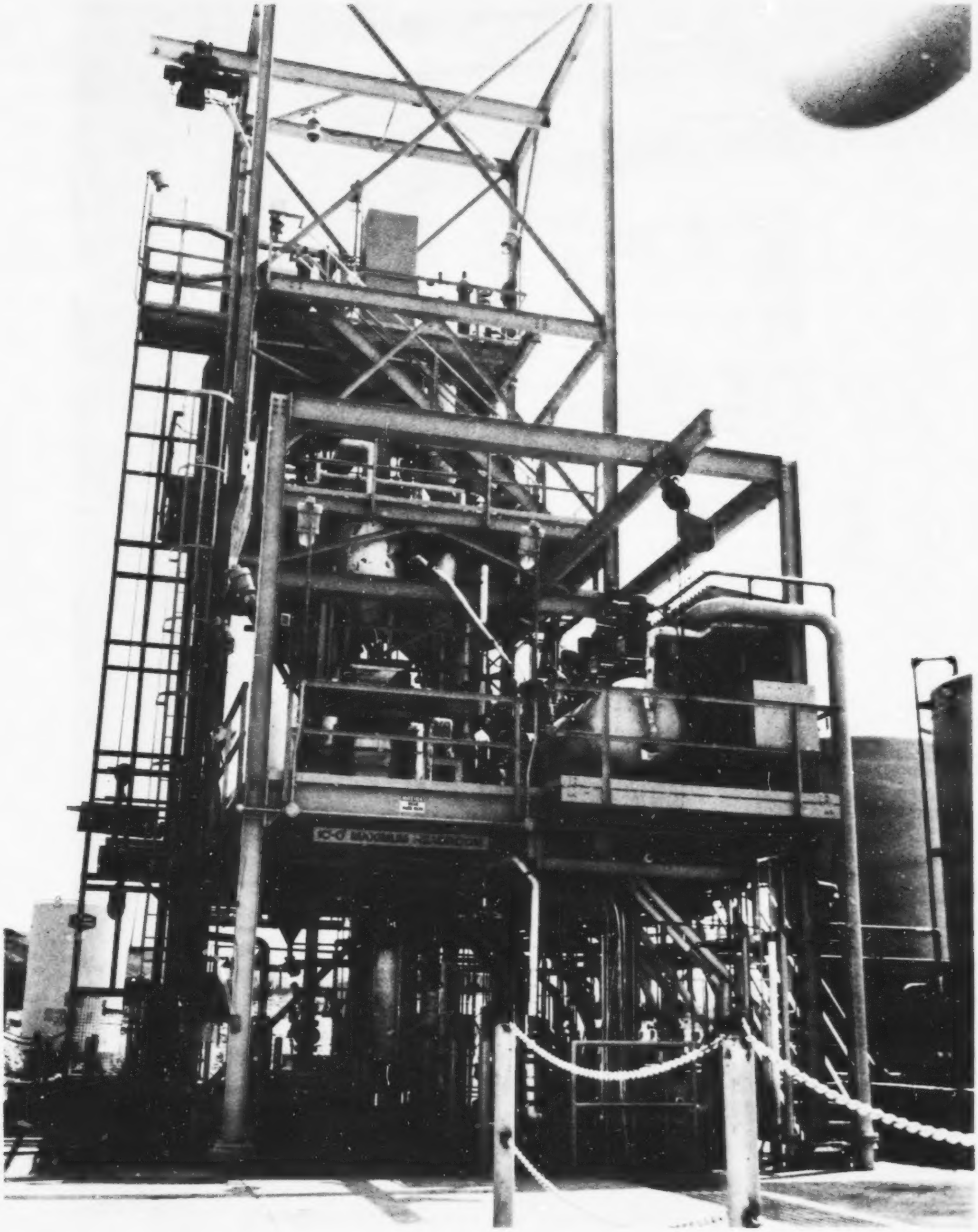


Figure 5. VIEW RTU - West Side.

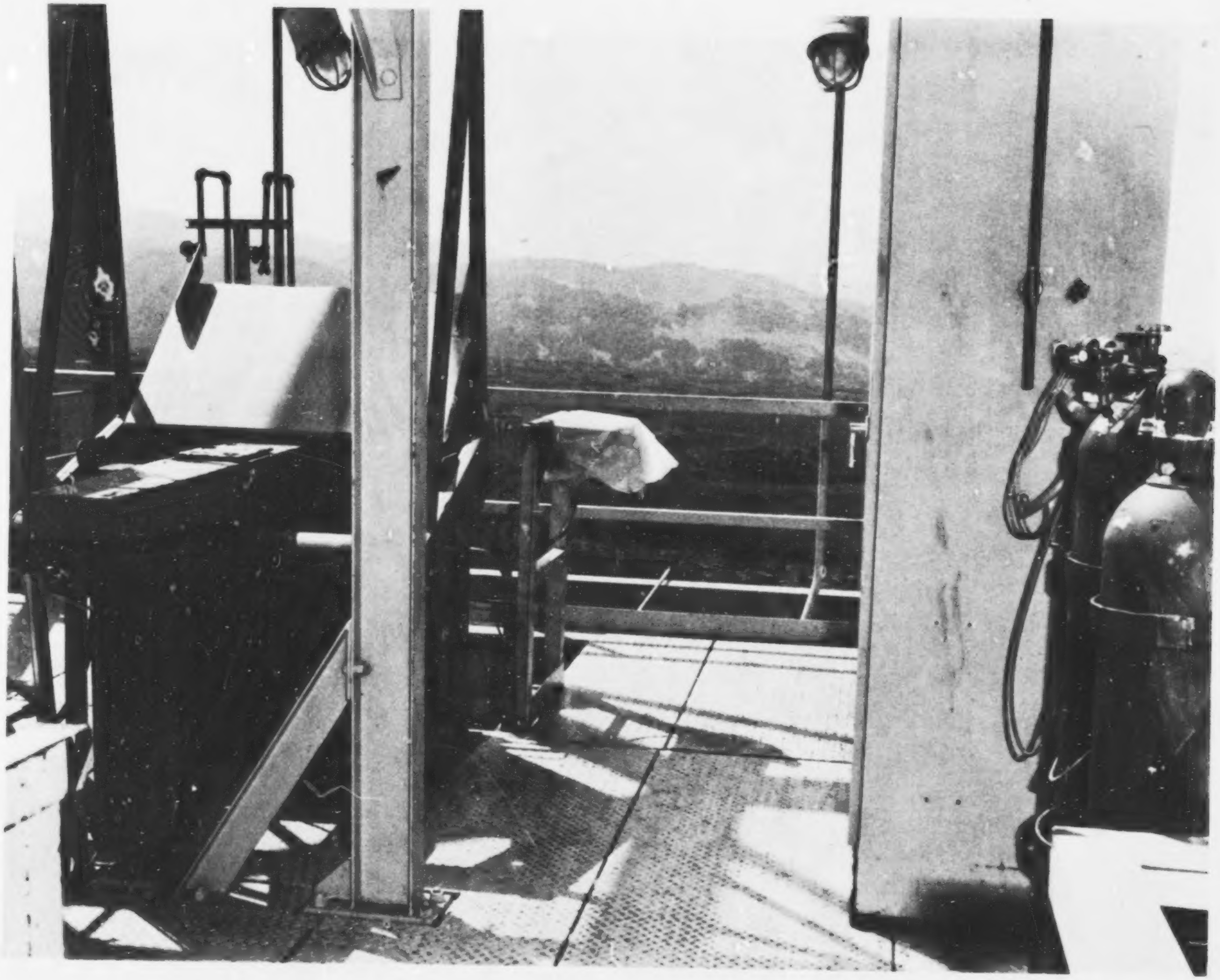


Figure 6. Coal Bin Tilter and Oxygen Analyzer.

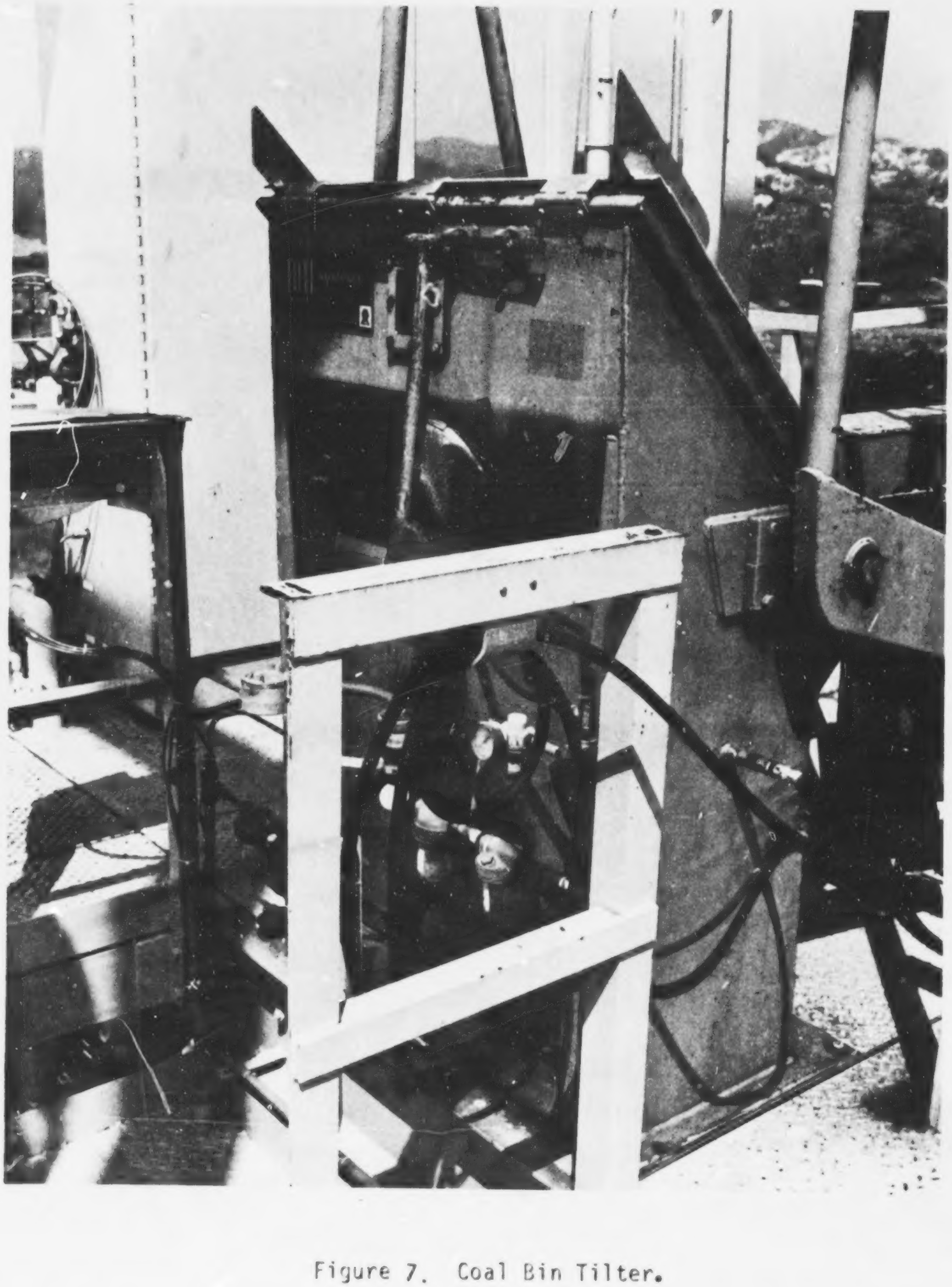


Figure 7. Coal Bin Tilter.

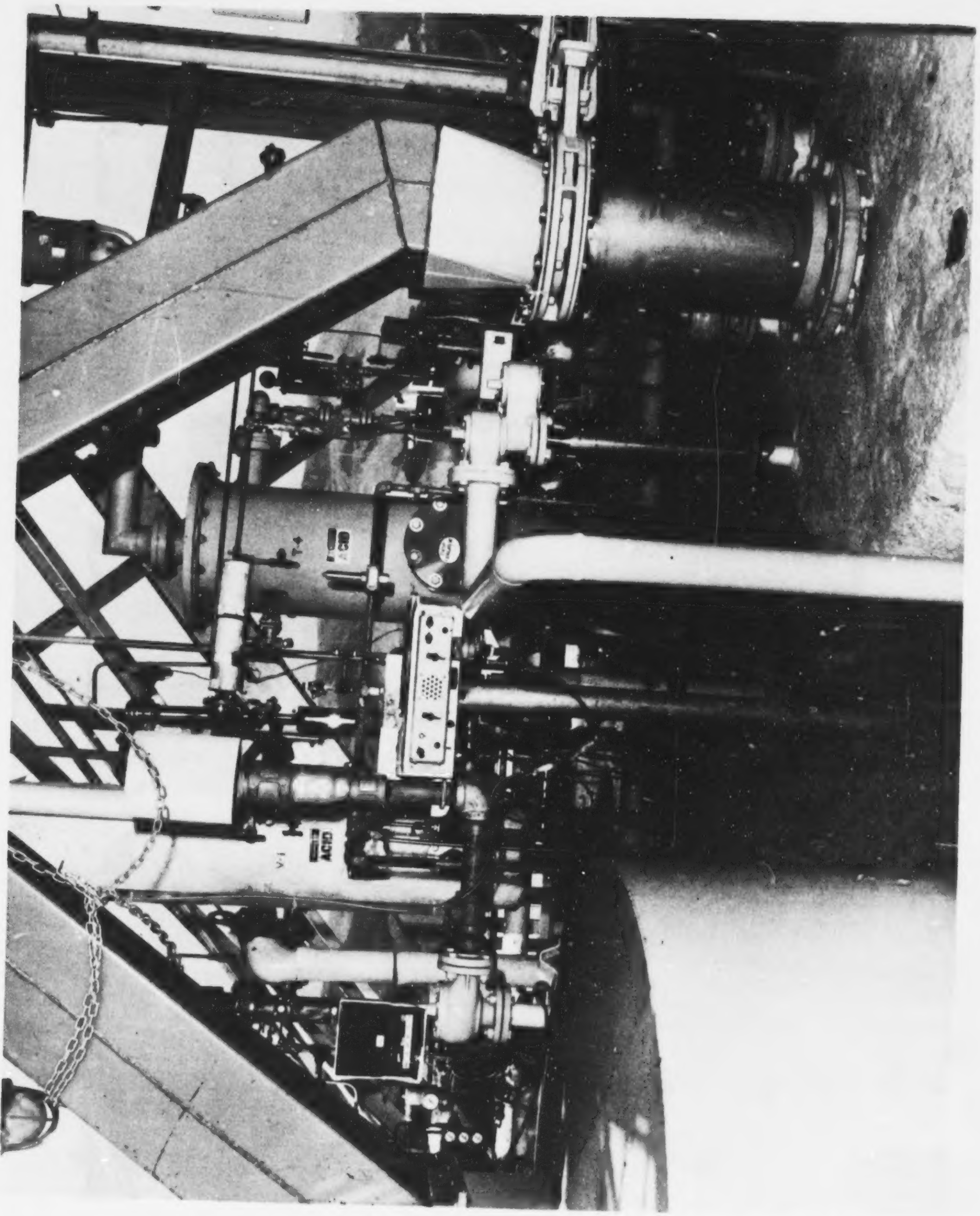

T-4
ACID
V-1
ACID

Figure 8. Equipment on Level 4 of RTU.

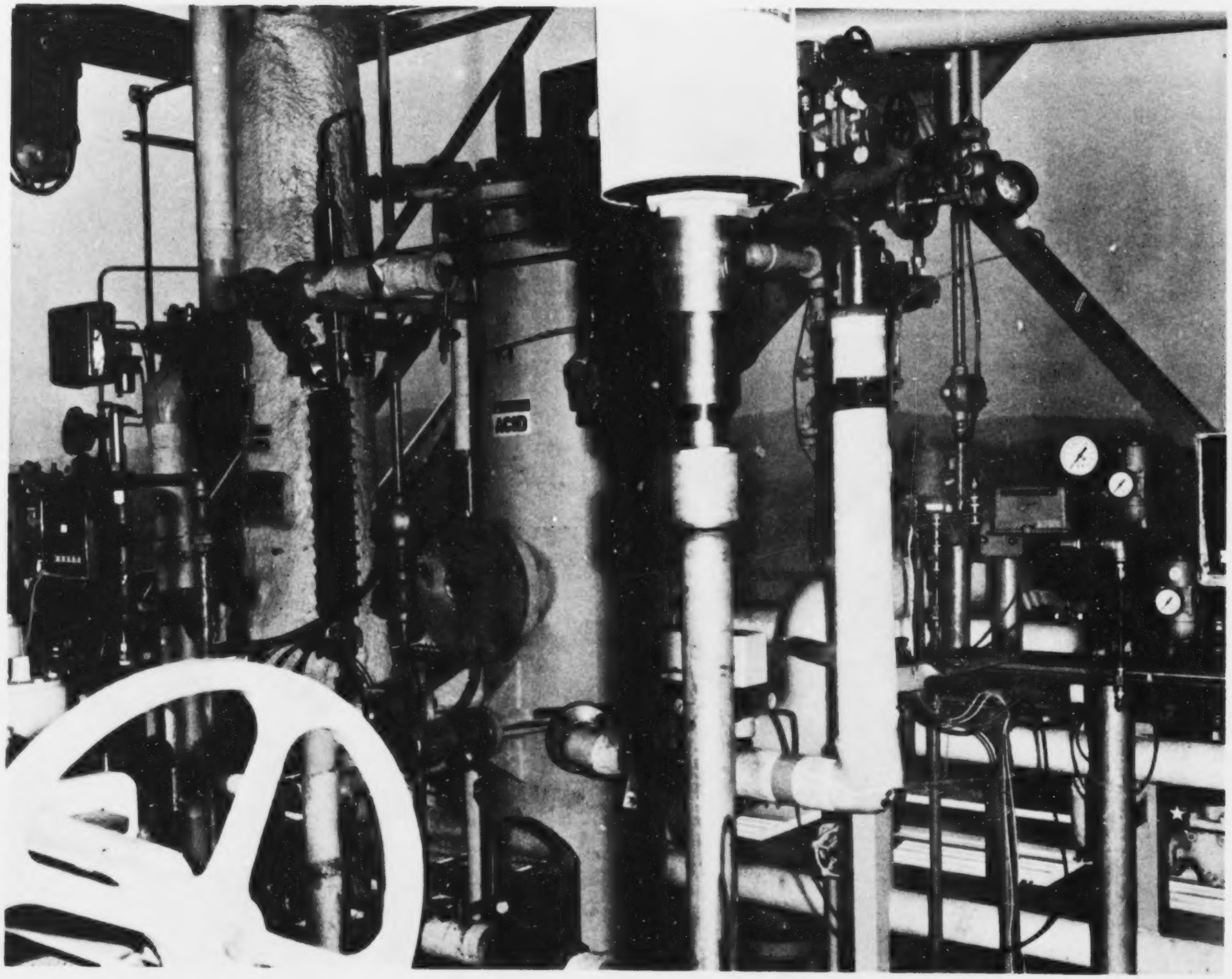


Figure 9. Vent Gas Scrubber T-4 and Knockout Drum V-1.

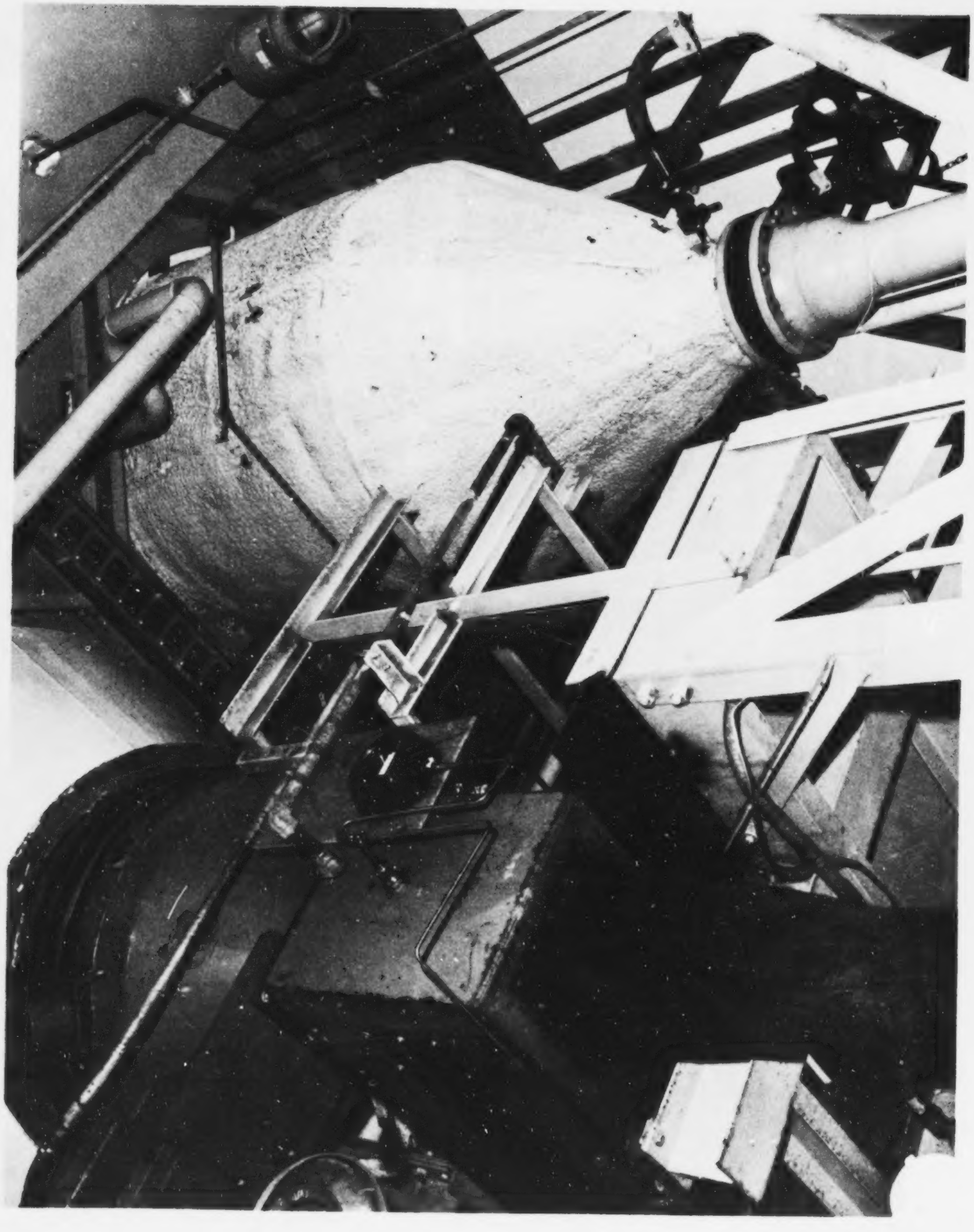


Figure 10. Coal Feed Tanks T-1 and T-6 and Weigh Belt Feeder A-3.

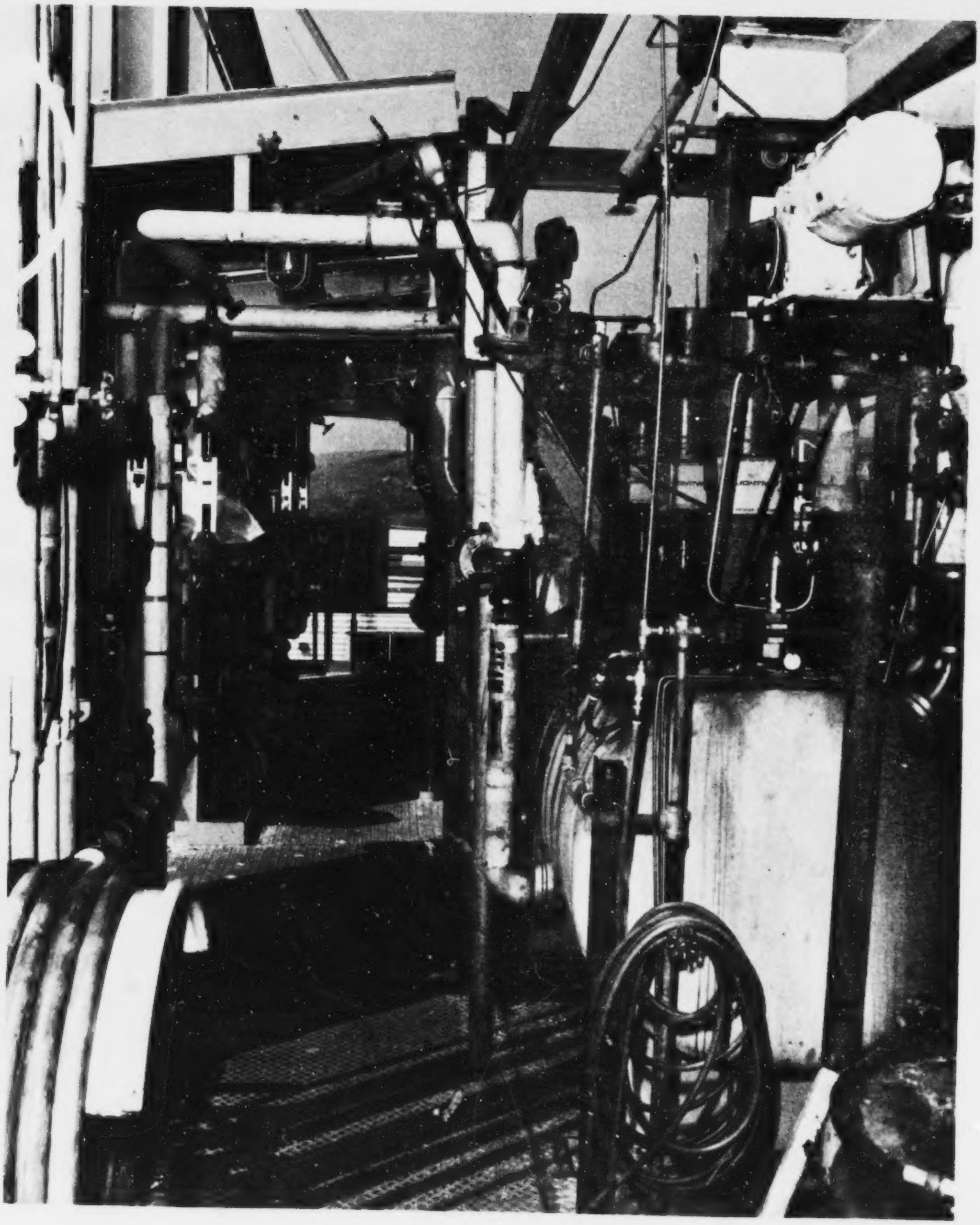


Figure 11 Slurry Mix Tank T-2.

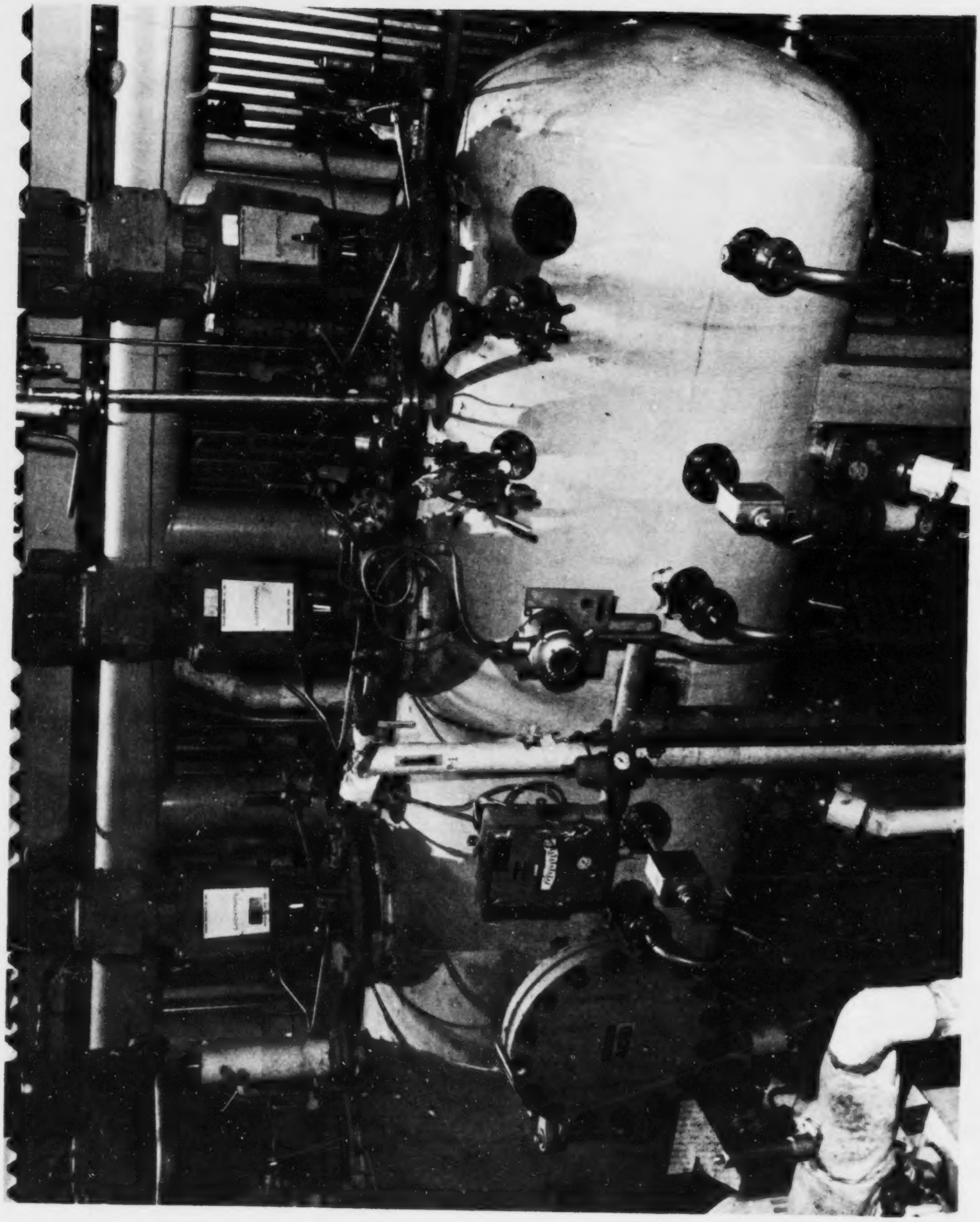


Figure 12. Primary Reactor R-1.

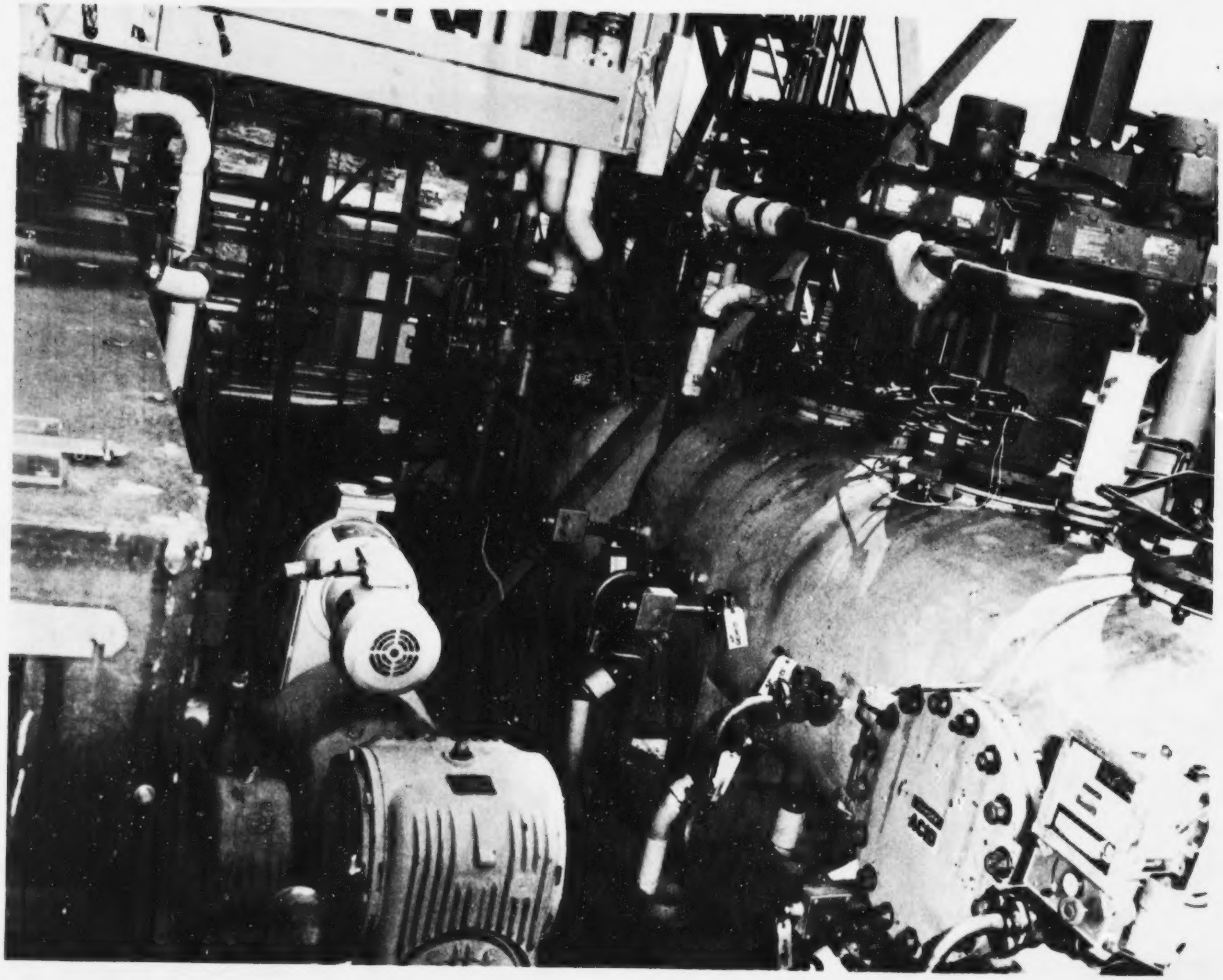


Figure 13. Slurry Mix Tank T-2 and Primary Reactor R-1.

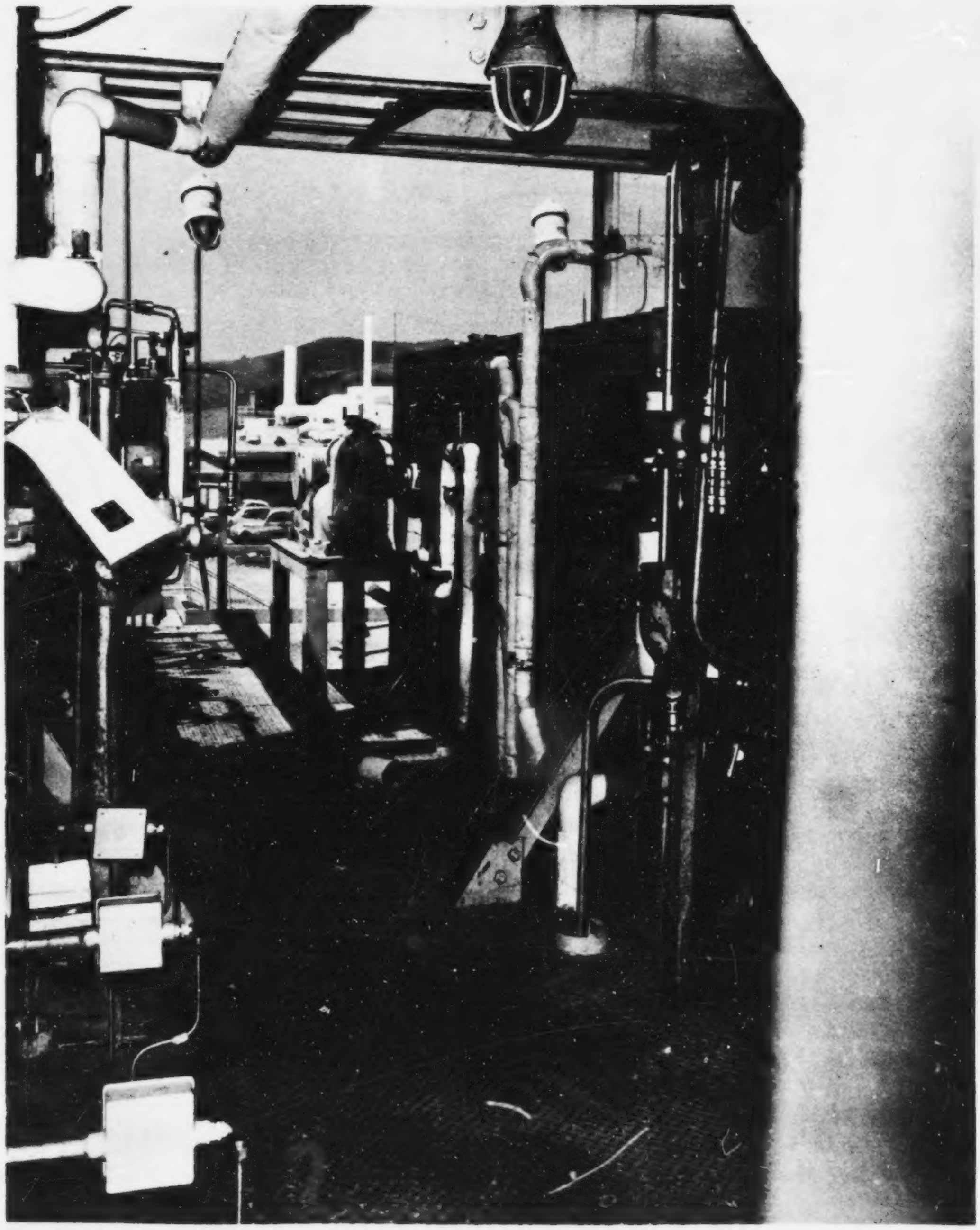


Figure 14. Belt Filter.

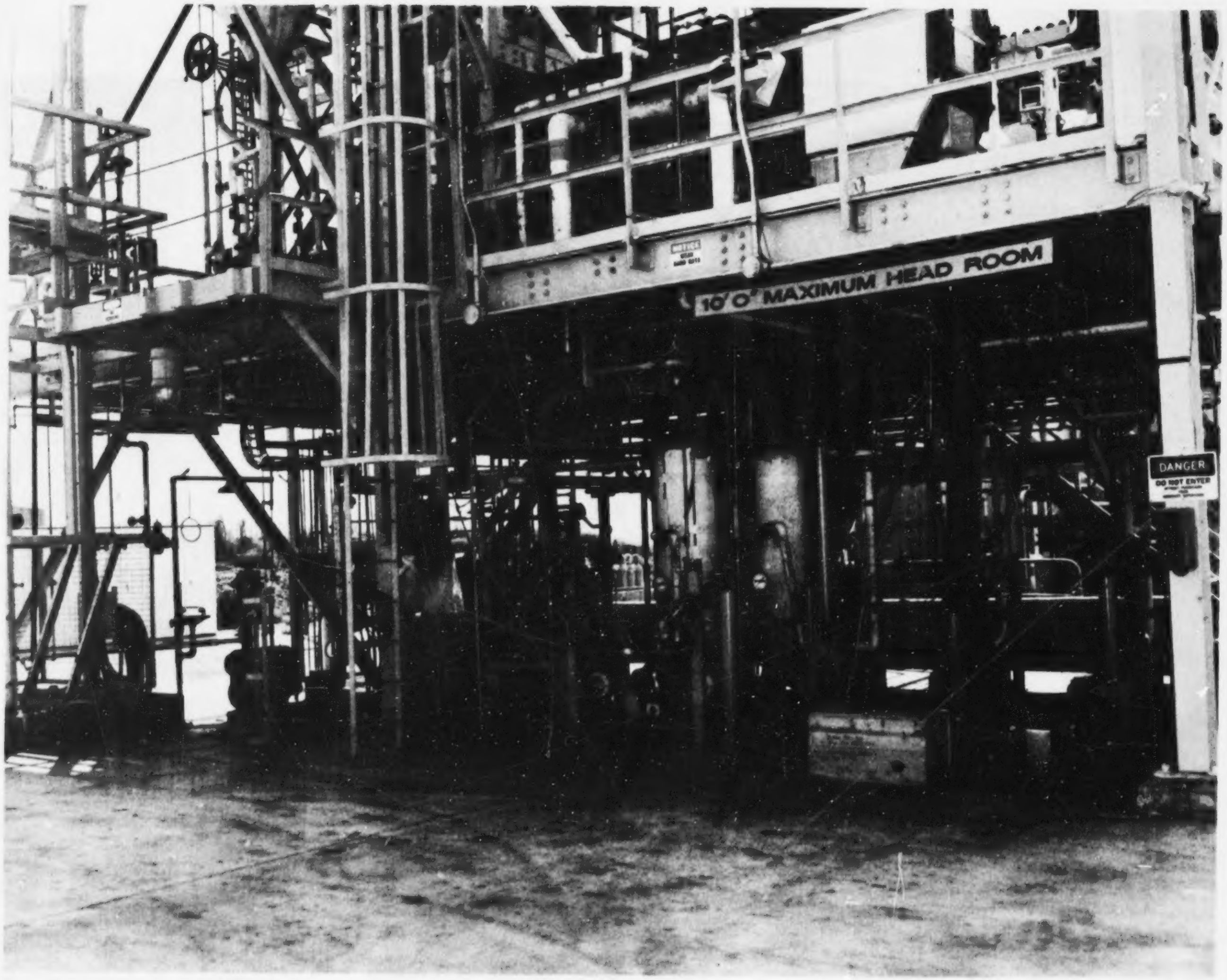

10'0" MAXIMUM HEAD ROOM
DANGER
DO NOT ENTER

Figure 15. Filtrate Collection Equipment.

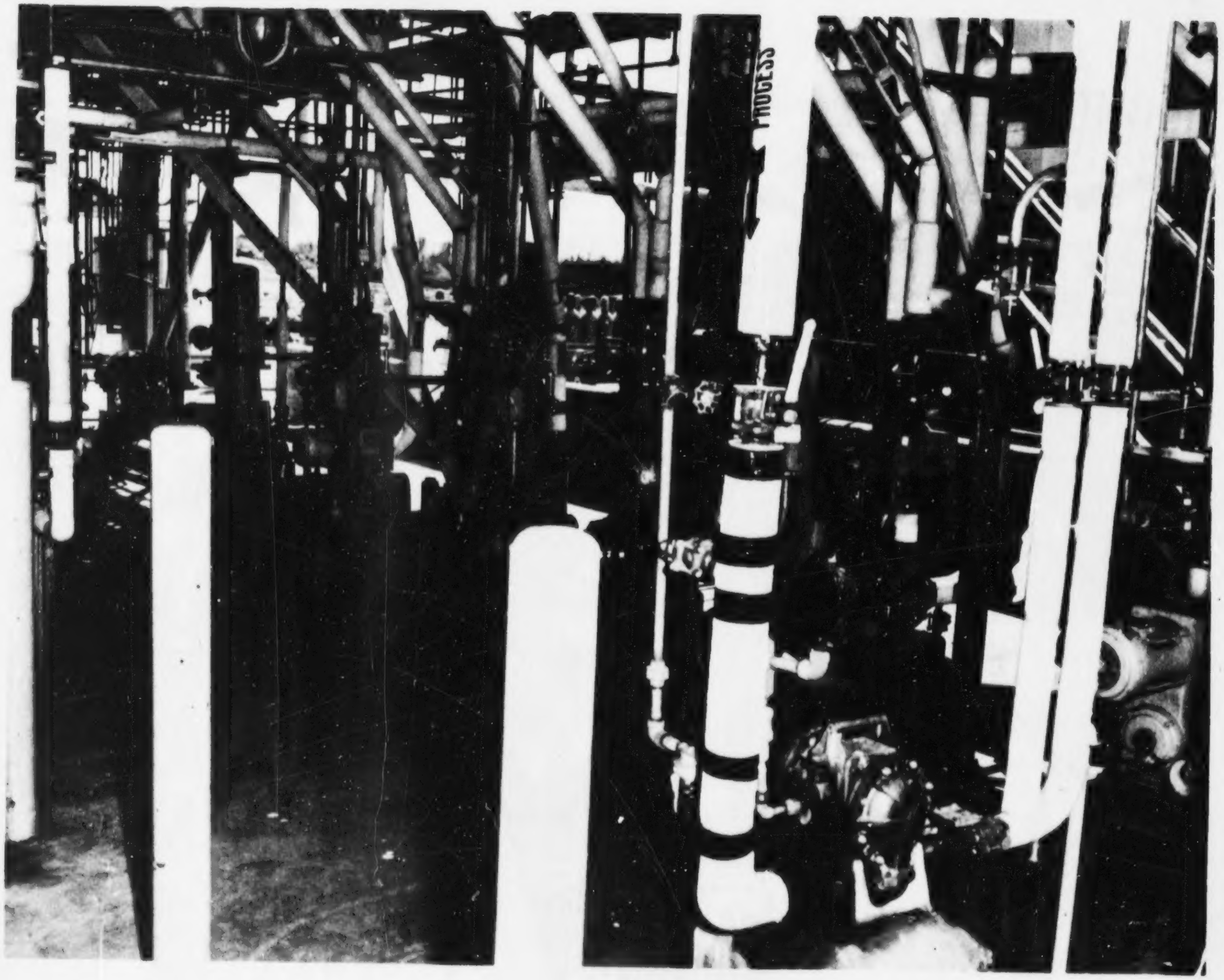


Figure 16. Recirculation Pumps for Reactor R-1.

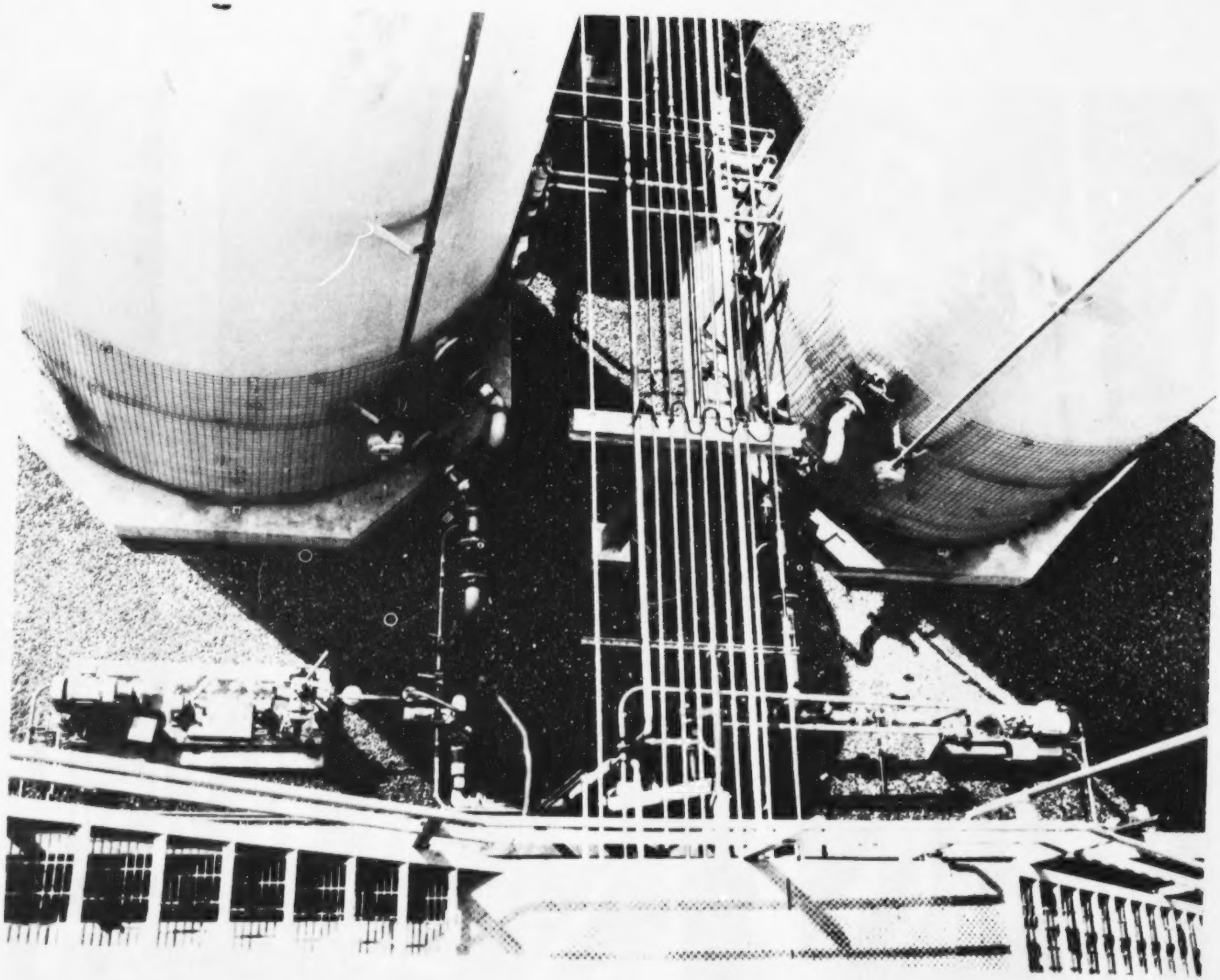


Figure 17. Leach Solution Storage Tanks and Pumps.

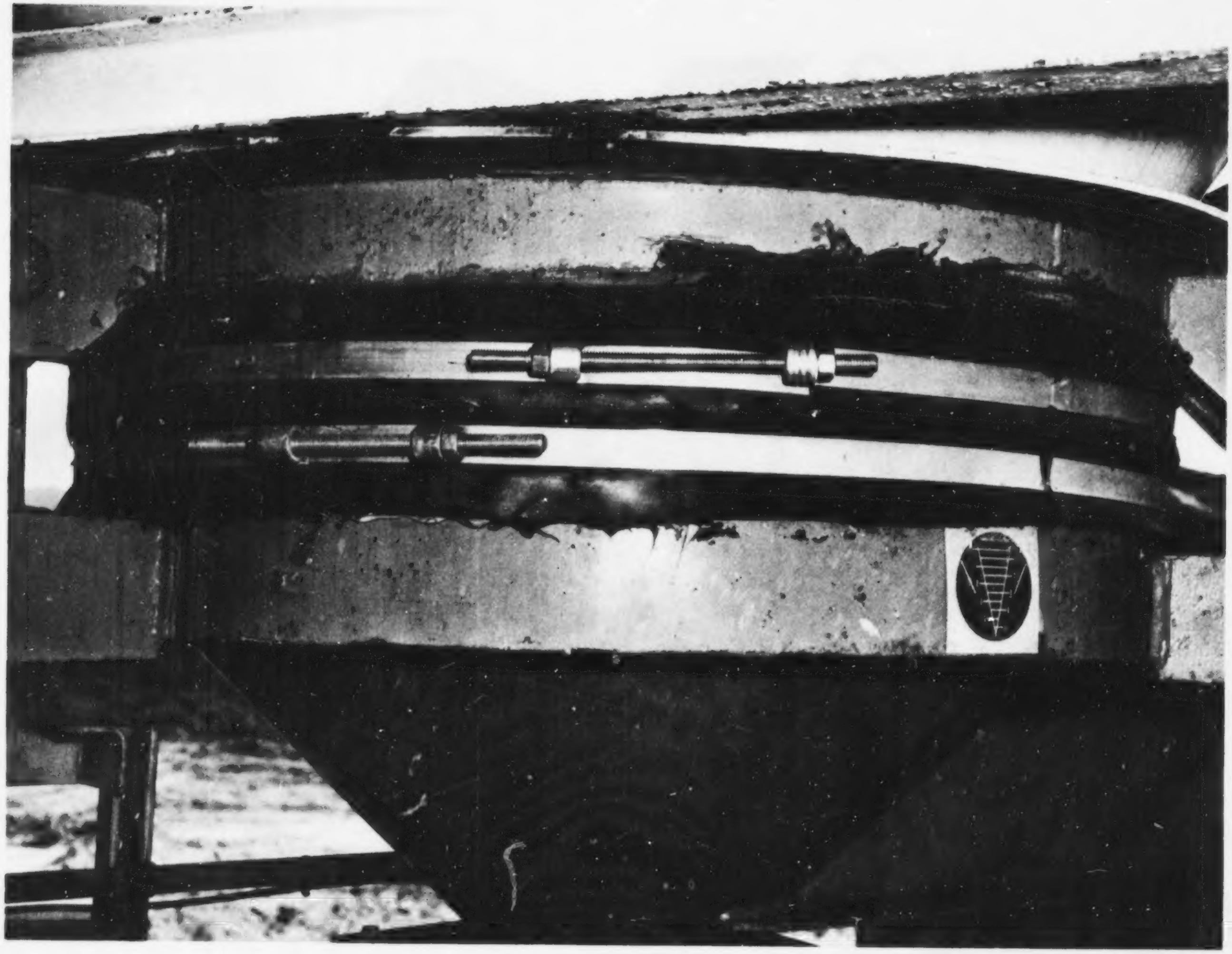


Figure 18. Rubber Boot Between Coal Storage Tank T-1 and Live Bottom Feeder A-2.

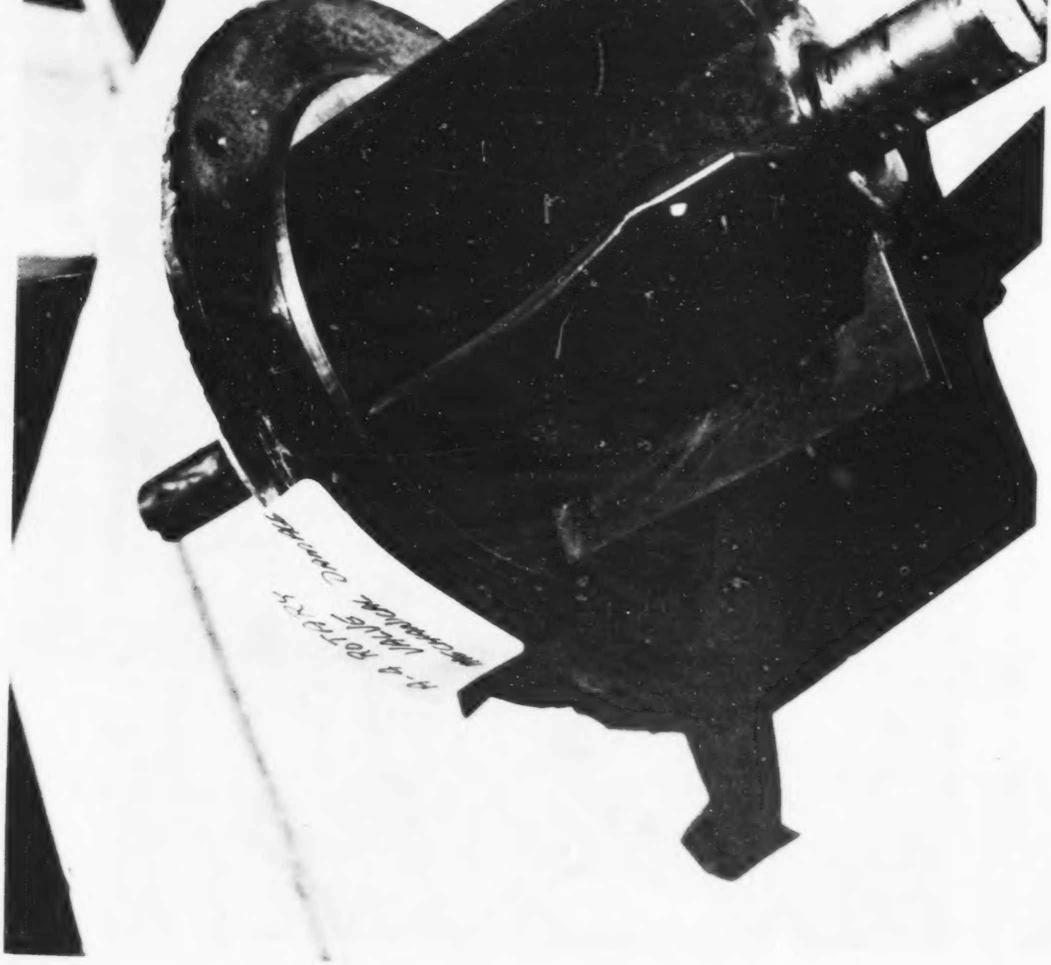

A-4 ROTORS
VALVE
Mechanical Damage

Figure 19. Rotory Vane from Coal Feed Valve A-4.

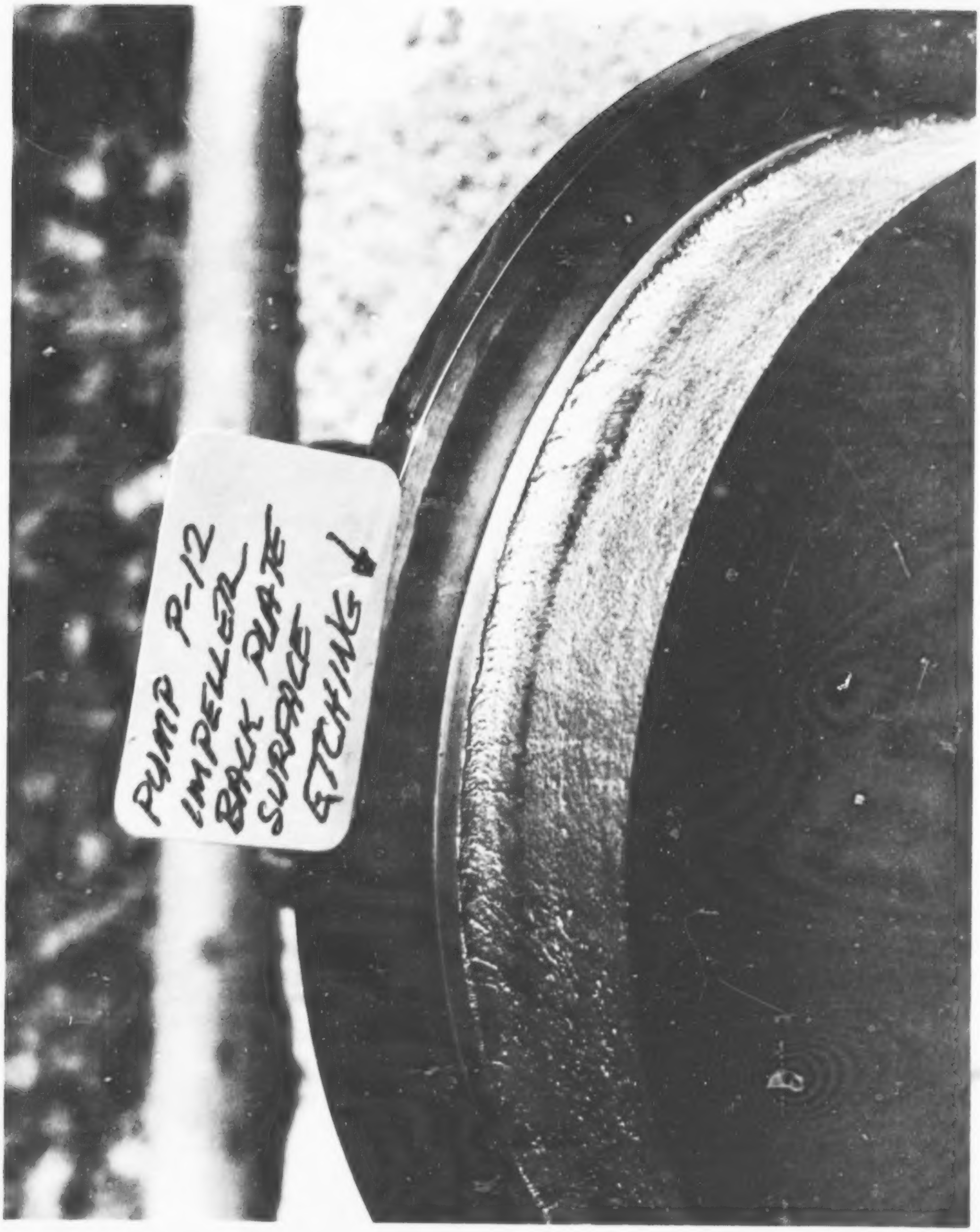

PUMP P-12
IMPELLER
BACK PLATE
SURFACE
ETCHING

Figure 20. Impeller Back Plate from Leach Solution Circulation Pump P-12.

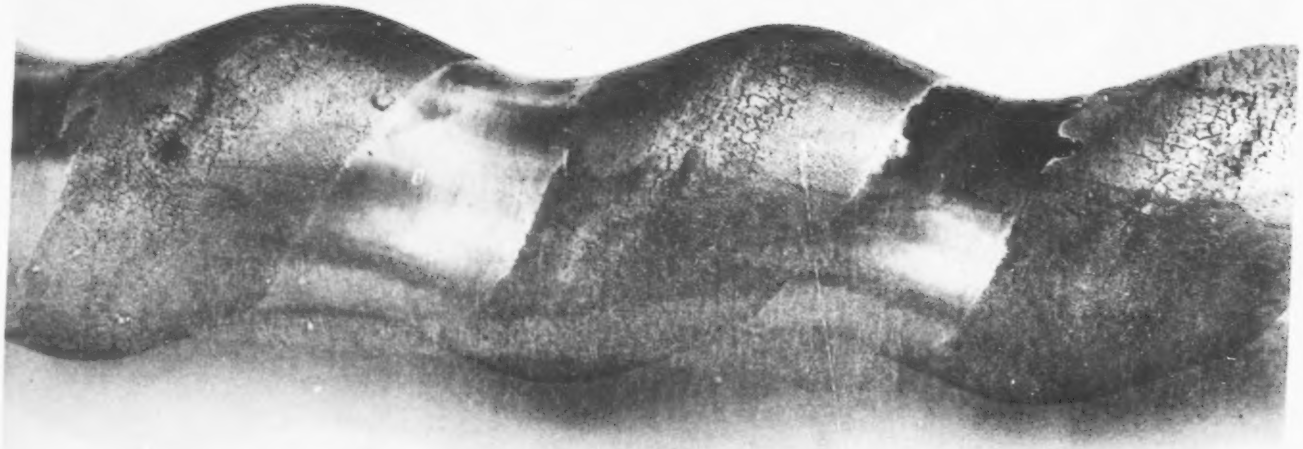


Figure 21. Rotor from Leach Solution Feed Pump P-13.

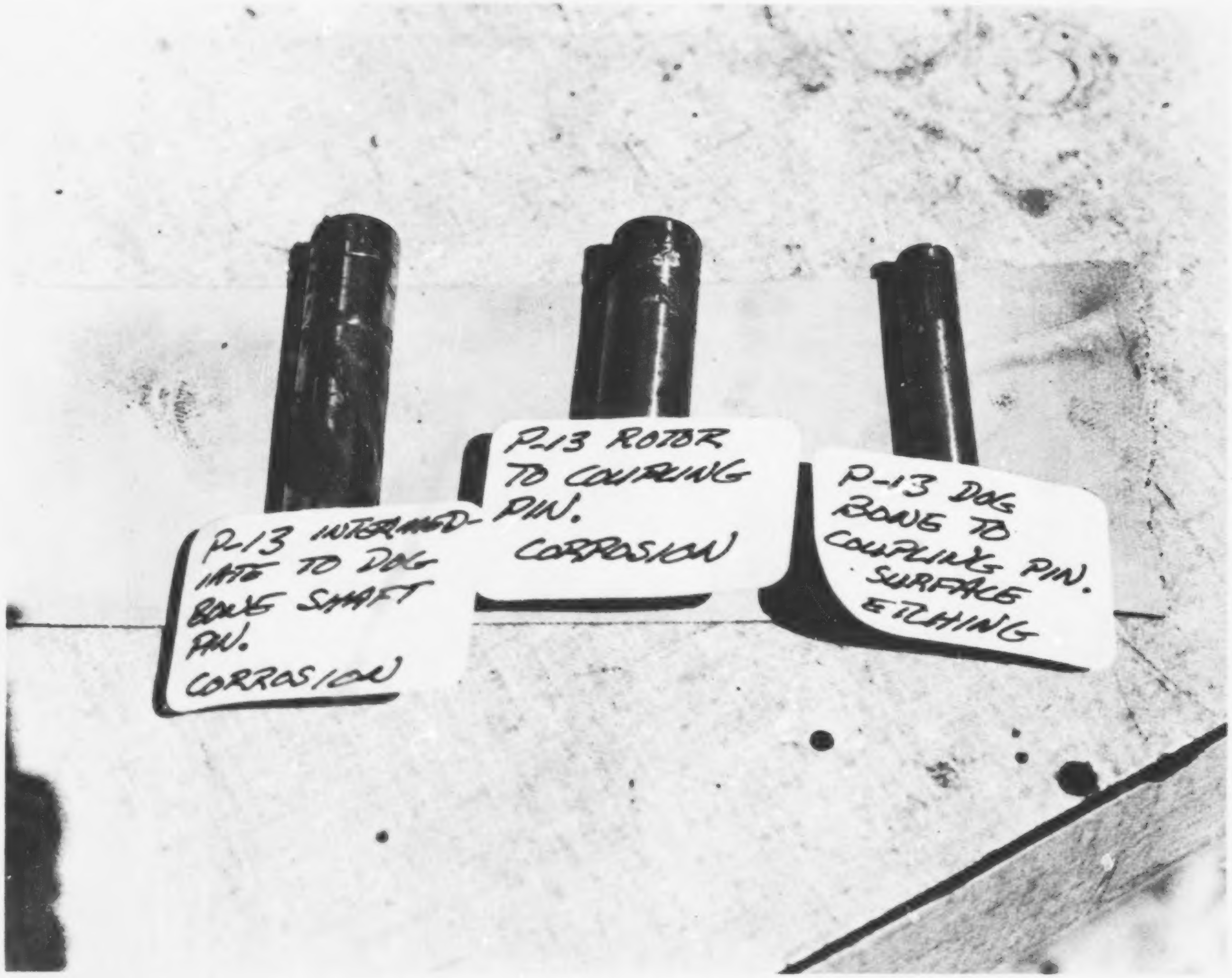

P-13 INTERMED-IATE TO DOG BONE SHAFT PIN. CORROSION
P-13 ROTOR TO COUPLING PIN. CORROSION
P-13 DOG BONE TO COUPLING PIN. SURFACE ETCHING

Figure 22. Shaft Pins from Leach Solution Feed Pump P-13.

V-1
BUBBLE CAP
TRAY

Figure 23. Bubble Cap Tray Inside Knockdown Drum V-1.

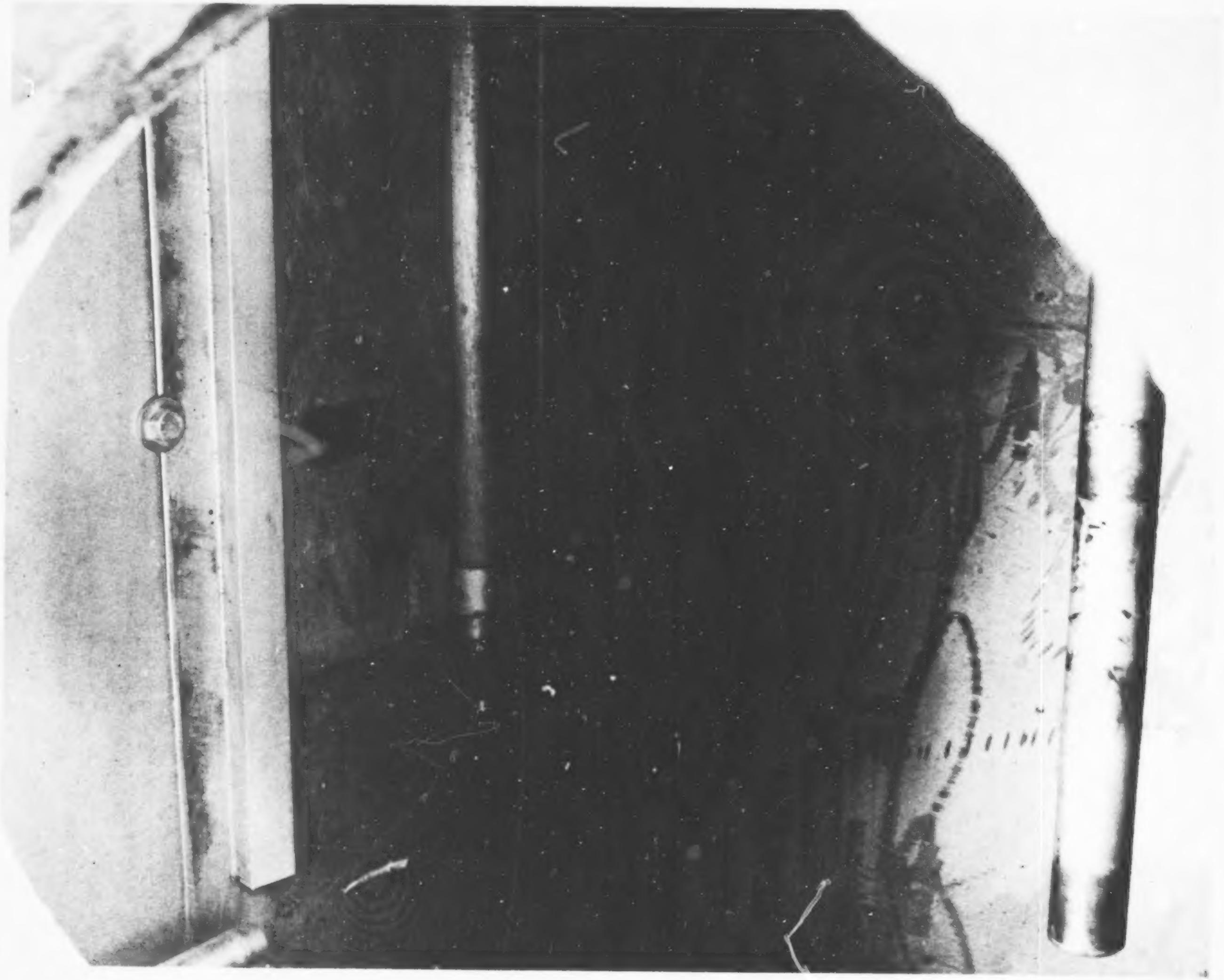


Figure 24. Weir Between Cells 1 and 2 of Mix Tank T-2.

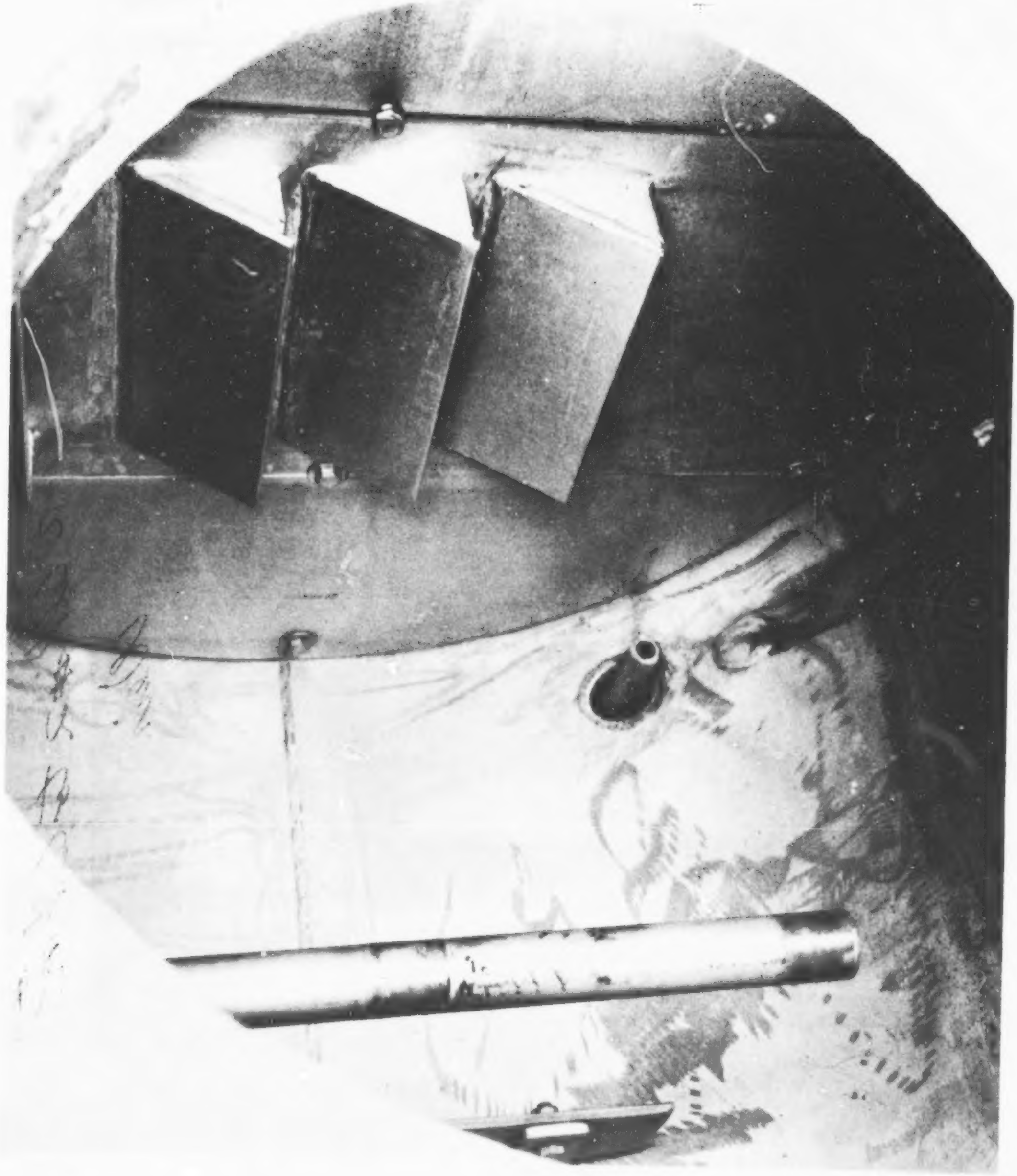


Figure 25. Weir Between Cells 2 and 3 of Mix Tank T-2.

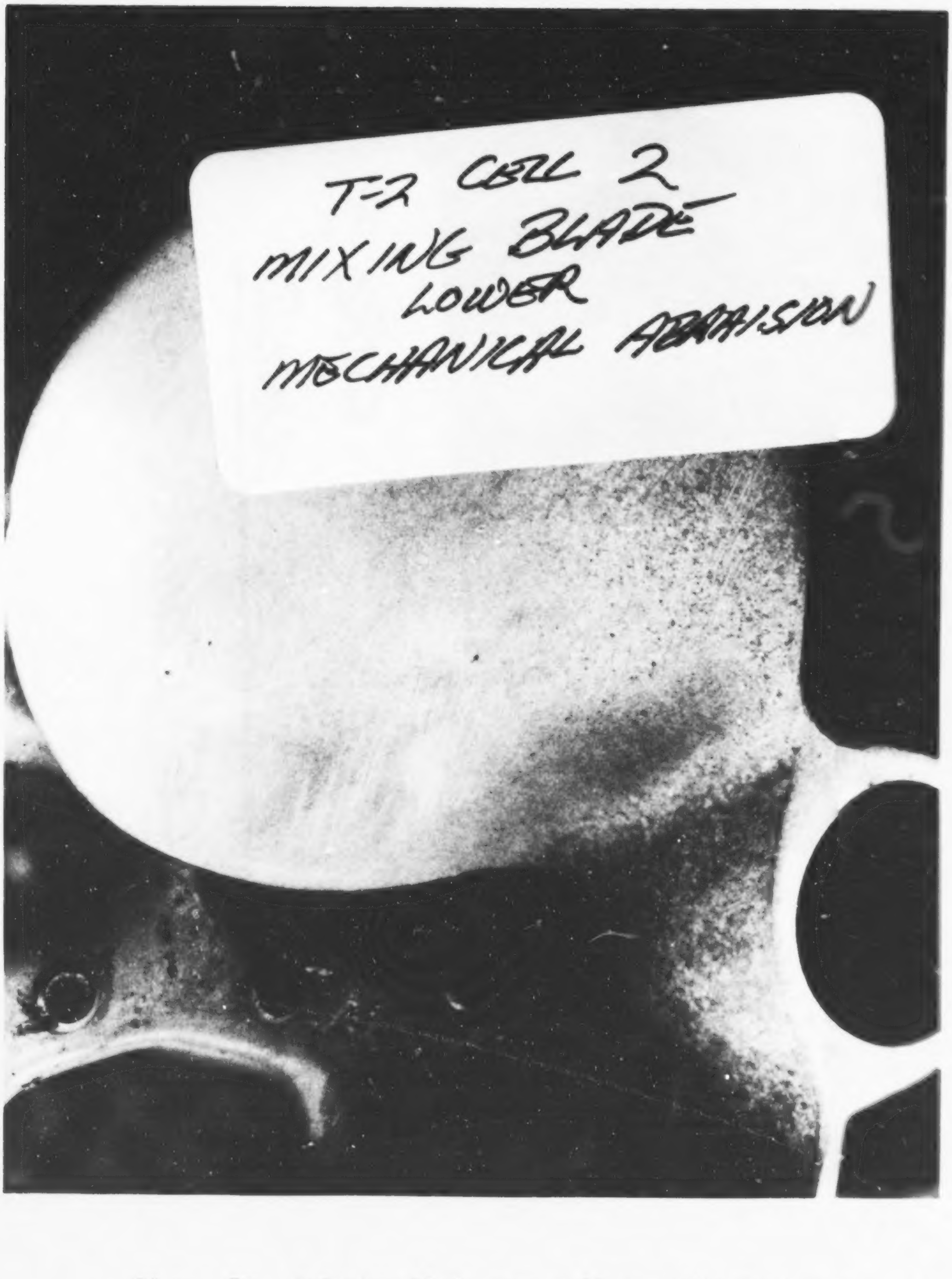
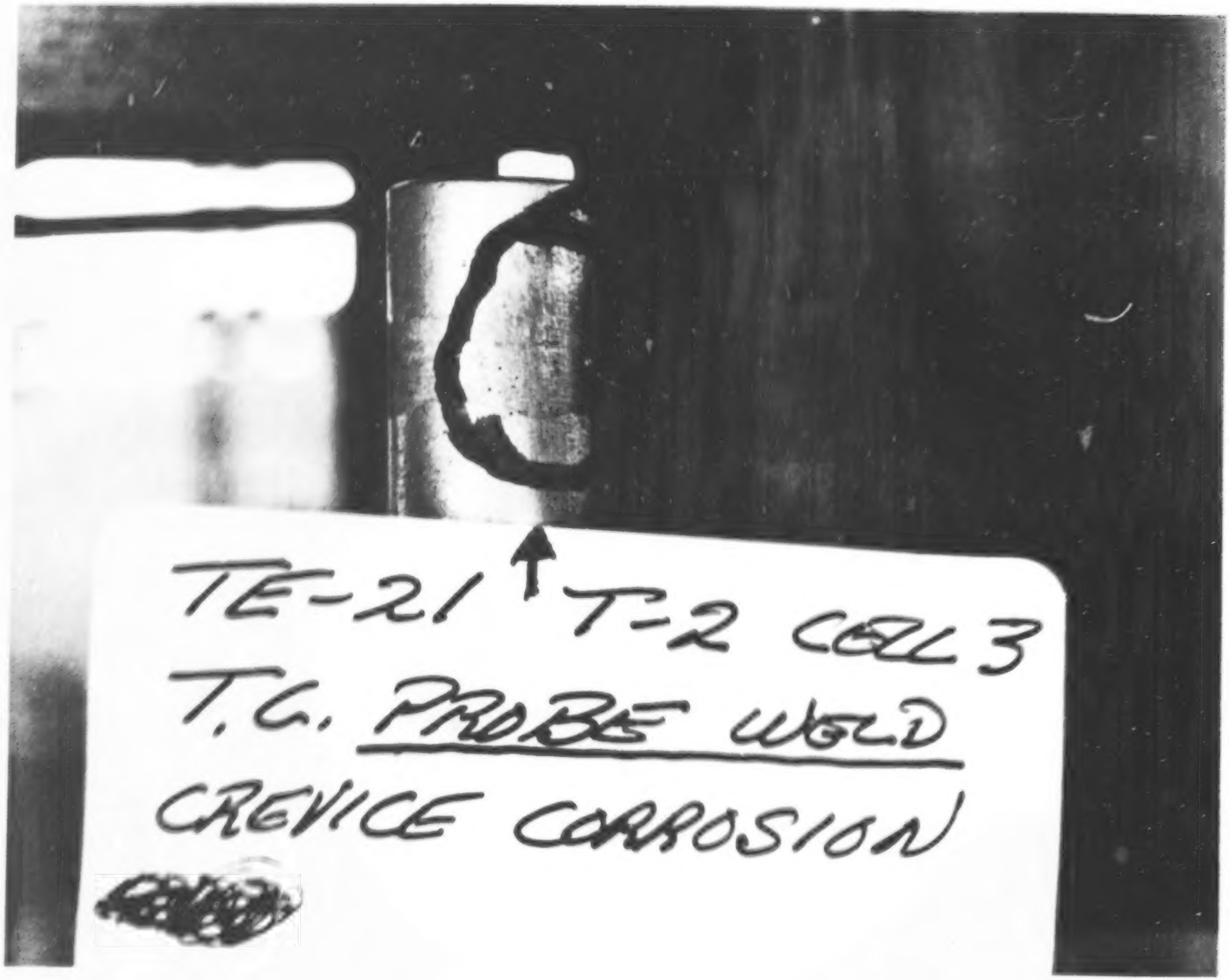

T-2 CELL 2
MIXING BLADE
LOWER
MECHANICAL ABRAISION

Figure 26. Agitator Blade from Cell 2 of Mix Tank T-2.



TE-21 T-2 CELL 3
T.C. PROBE WELD
CREVICE CORROSION

Figure 27. Thermocouple Probe TE-21 from Cell 3 of Mix Tank T-2.

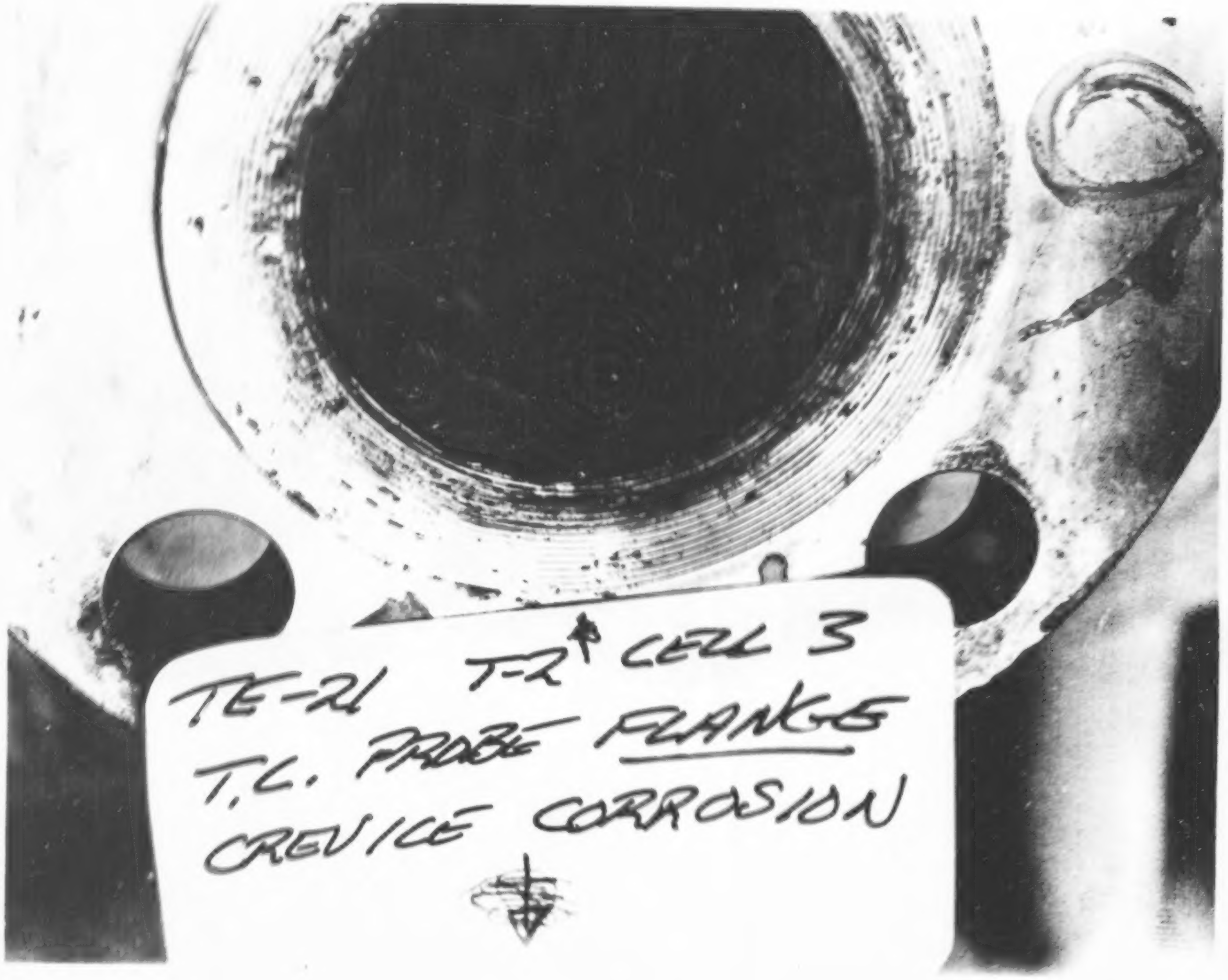

TE-21 T-2 CELL 3
T.C. PROBE FLANGE
CREVICE CORROSION

Figure 28. Thermocouple Well Flange in Cell 3 of Mix Tank T-2.

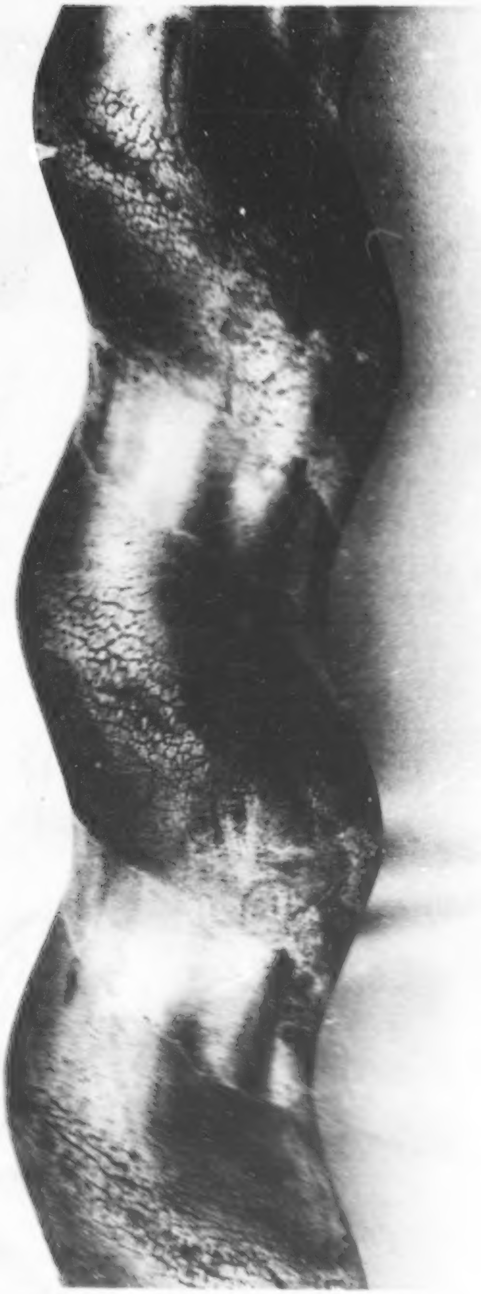


Figure 29. Rotor from Slurry Feed Pump R-1.

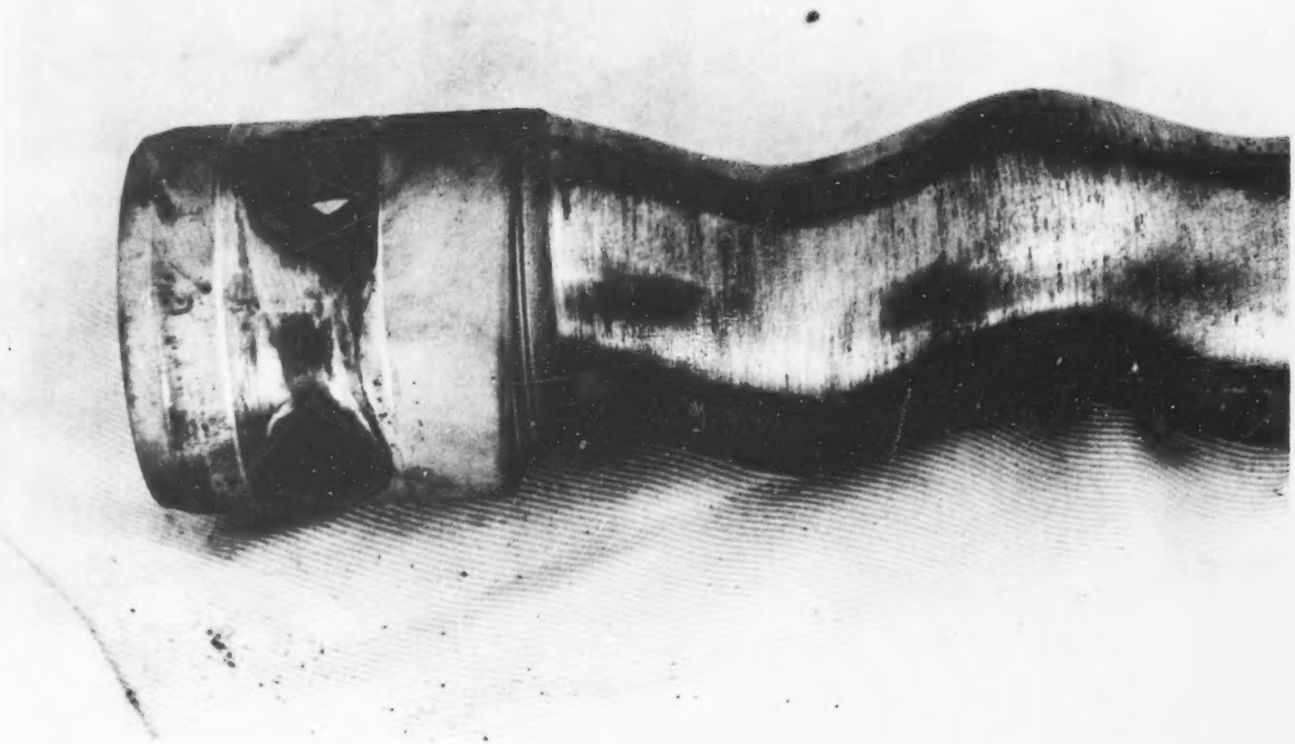


Figure 30. Rotor End from Slurry Feed Pump P-7.

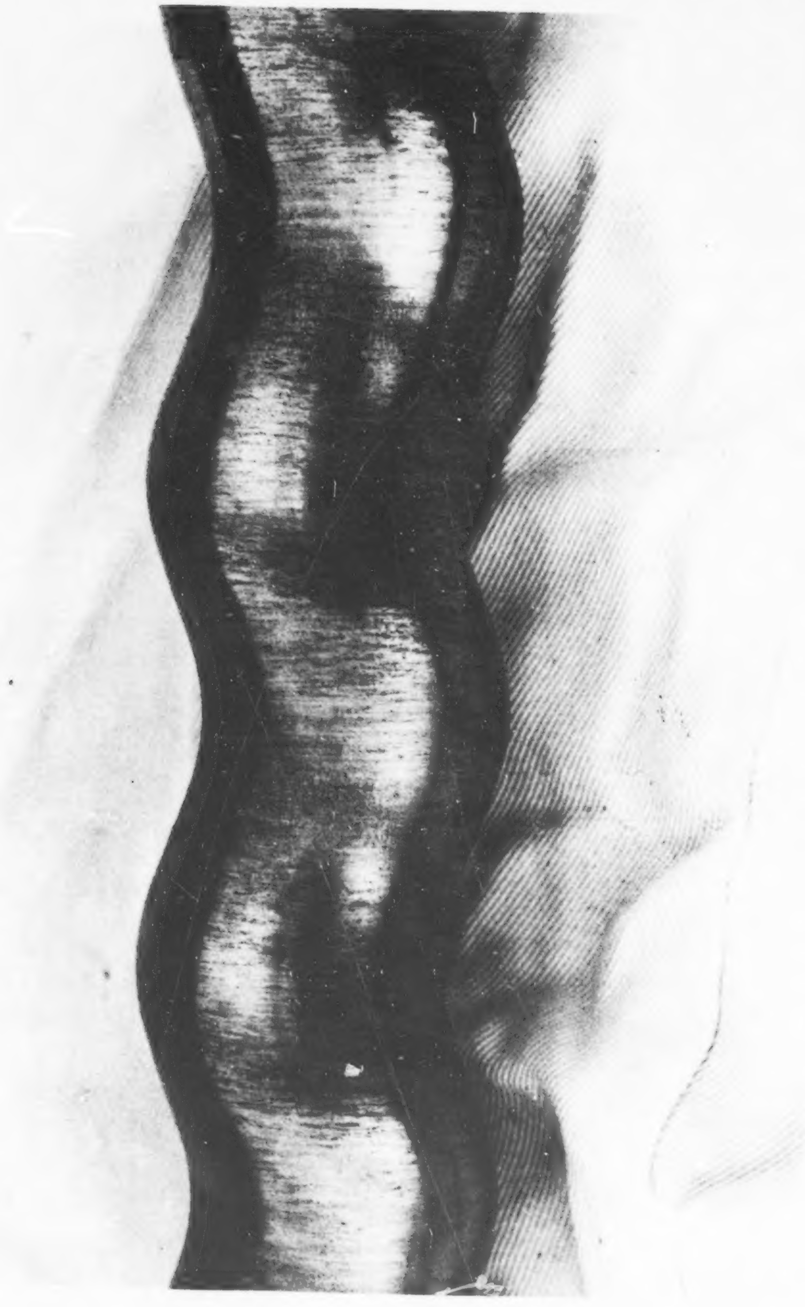


Figure 31. Rotor Center from Slurry Feed Pump P-7.

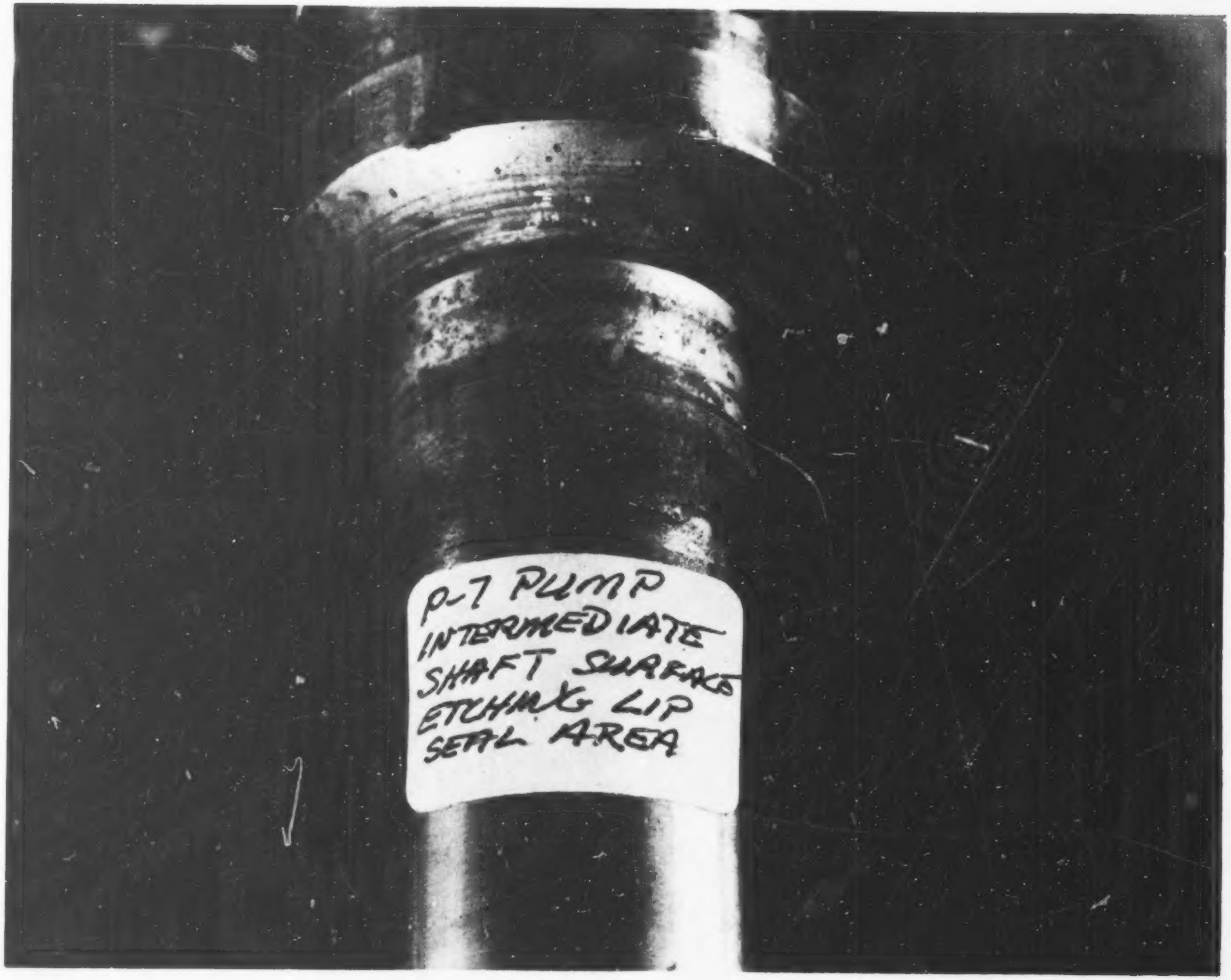

P-7 PUMP
INTERMEDIATE
SHAFT SURFACE
ETCHING LIP
SEAL AREA

Figure 32. Intermediate Drive Shaft from Slurry Feed Pump P-7.

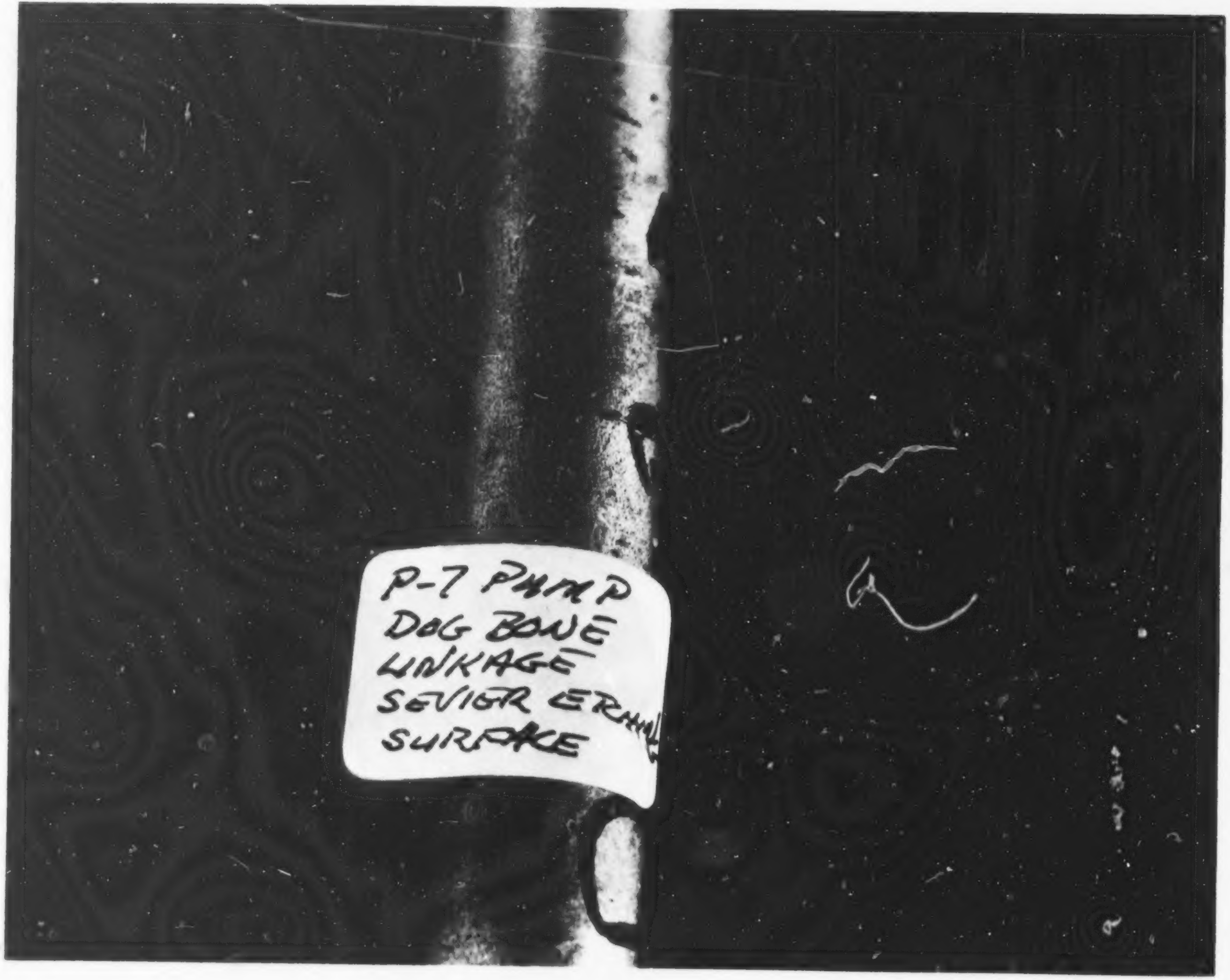

P-7 PUMP
DOG BONE
LINKAGE
SEVIOR ERAW
SURFACE

Figure 33. Universal Linkage Shaft from Slurry Feed Pump P-7.

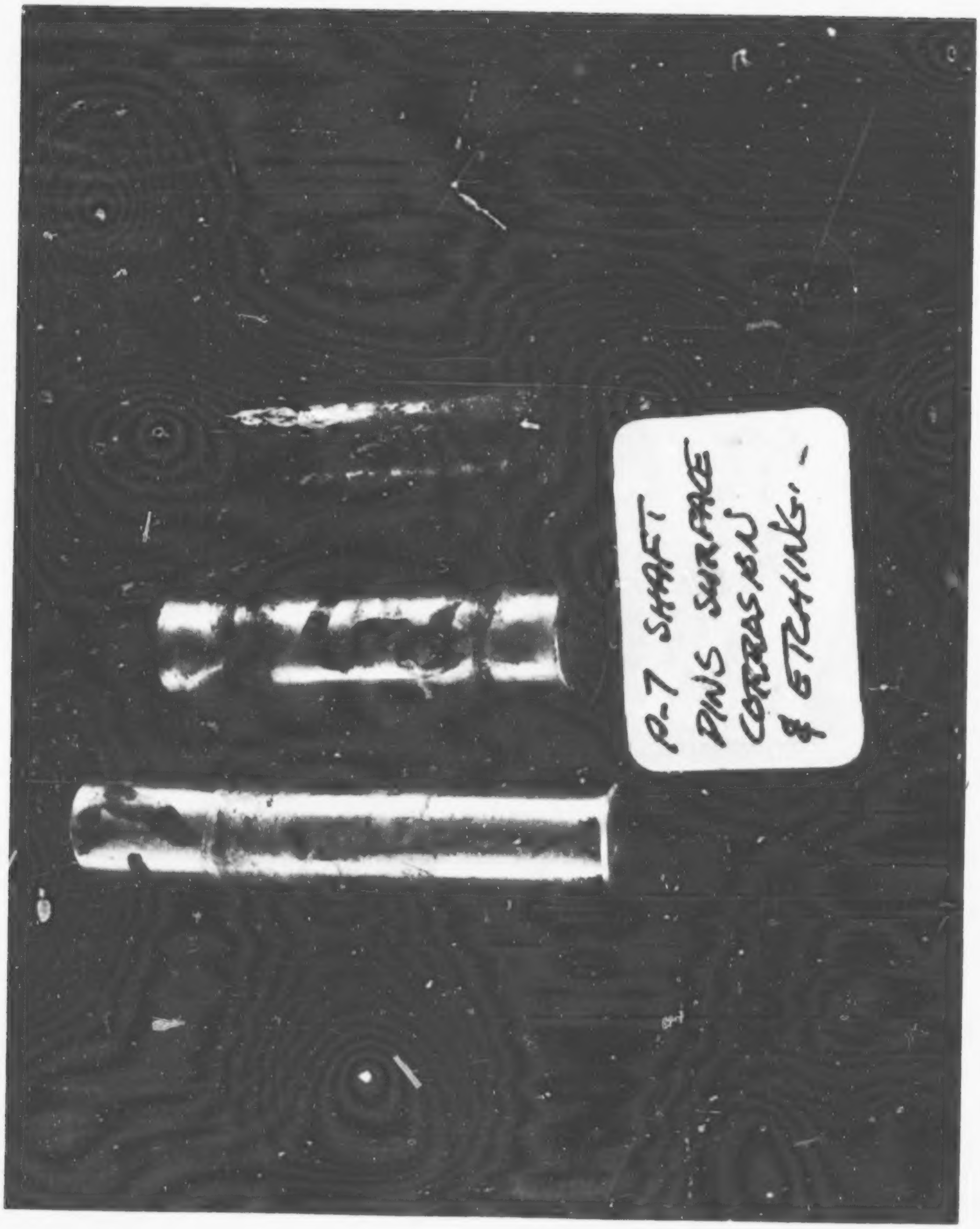

P-7 SHAFT
PINS SURFACE
CORROSION
& ETCHING.

Figure 34. Shaft Pins from Slurry Feed Pump P-7.

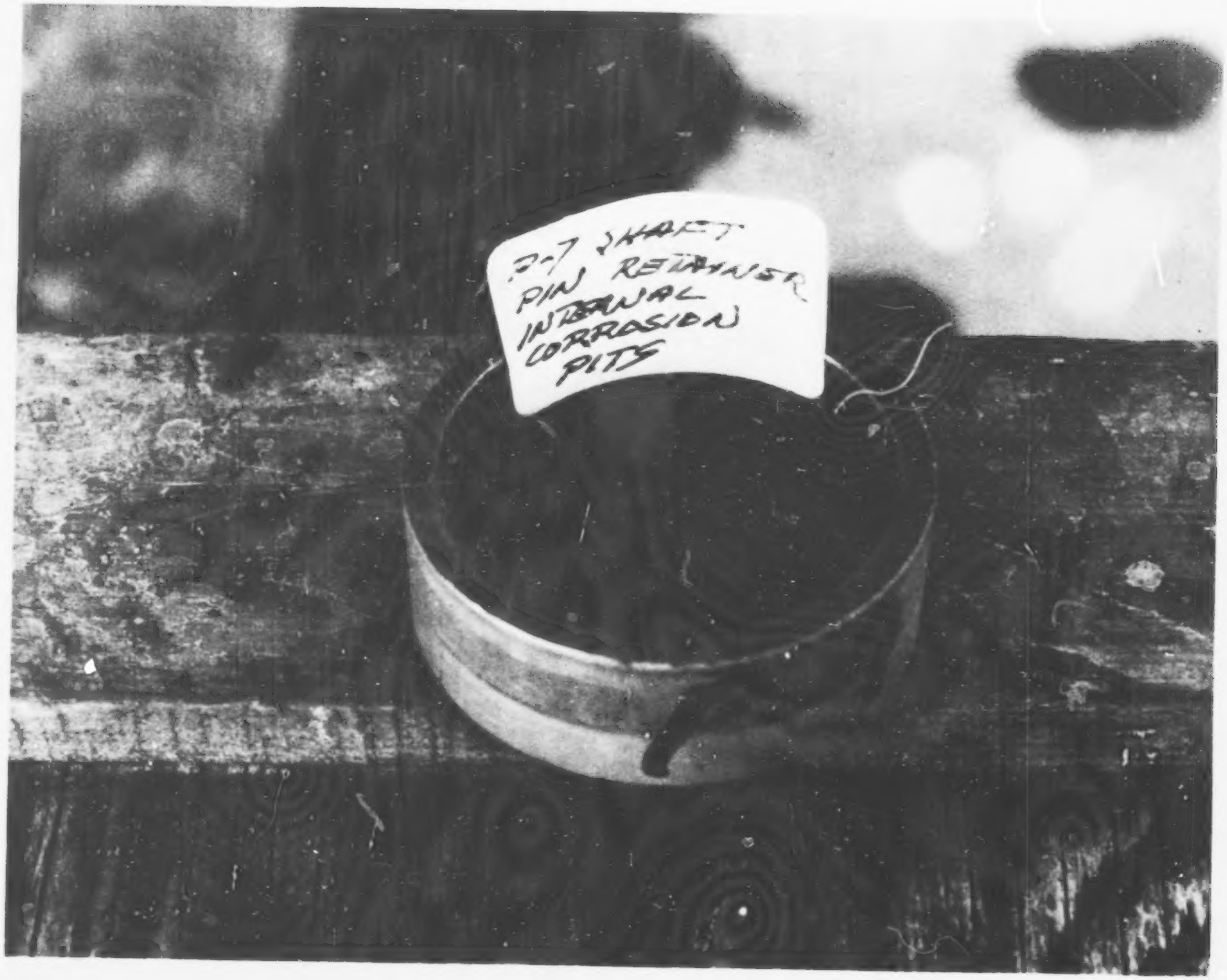

P-7 SHAFT
PIN RETAINER
INTERNAL
CORROSION
PITS

Figure 35. Retaining Ring from Slurry Feed Pump P-7.

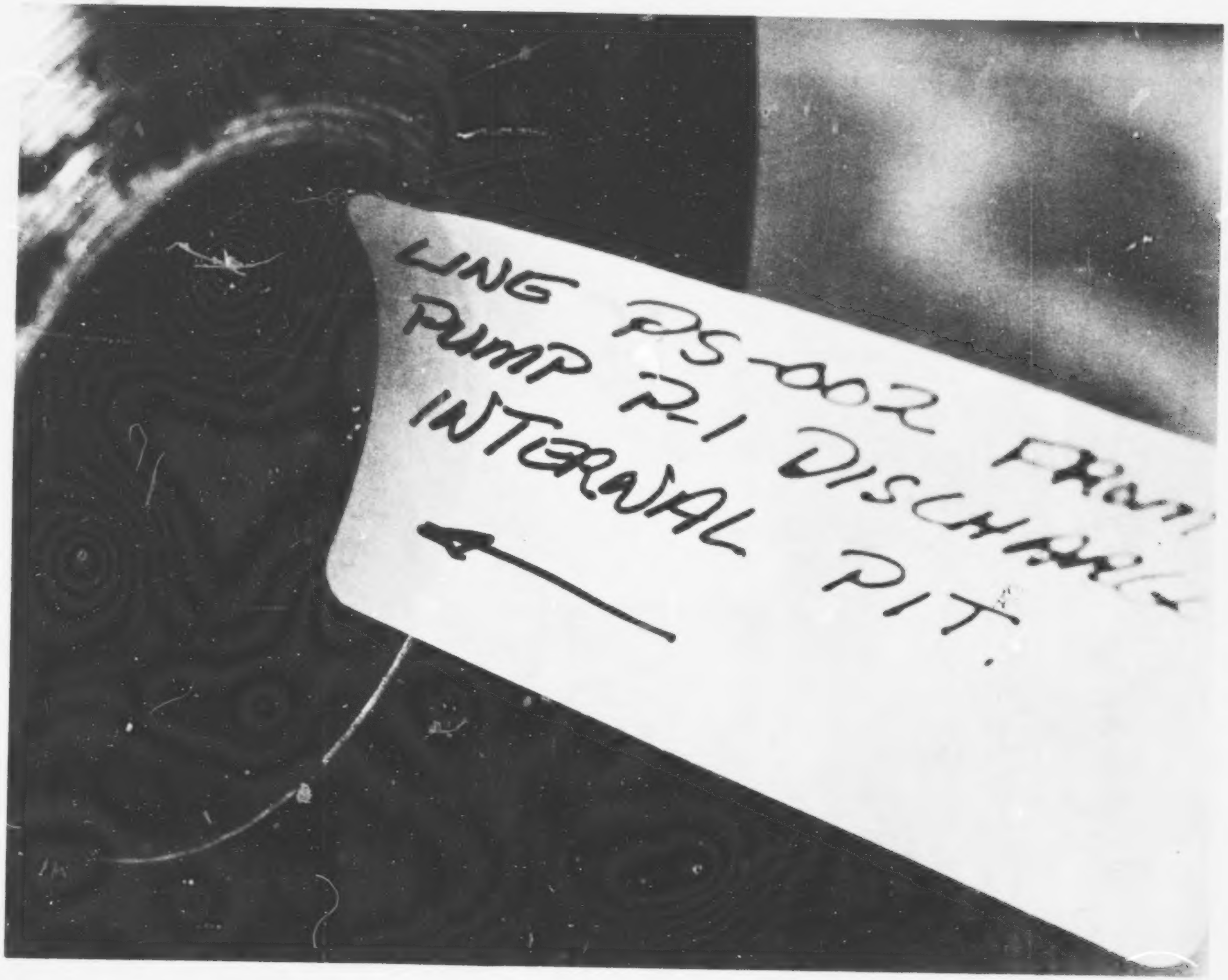

LING PS-002 FROM
PUMP P-1 DISCHARG
INTERNAL PIT.

Figure 36. Pipe Flange Which Mates with Slurry Feed Pump P-1 Discharge Port.

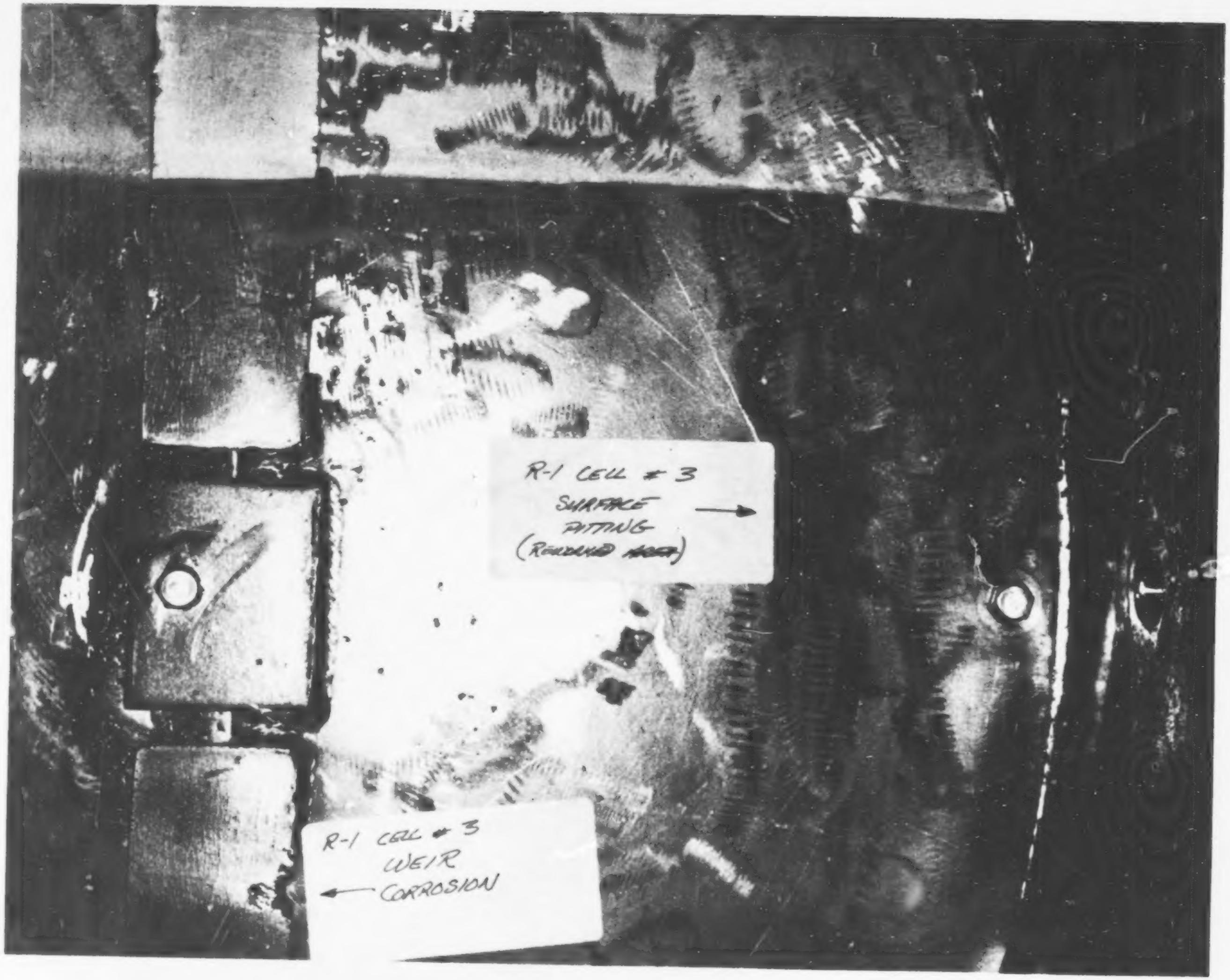

R-1 CELL # 3
SURFACE
PITTING
(REGROUND AREA)
R-1 CELL # 3
WEIR
CORROSION

Figure 37. Weir and Vessel Wall Inside Cell 3 of Primary Reactor R-1.

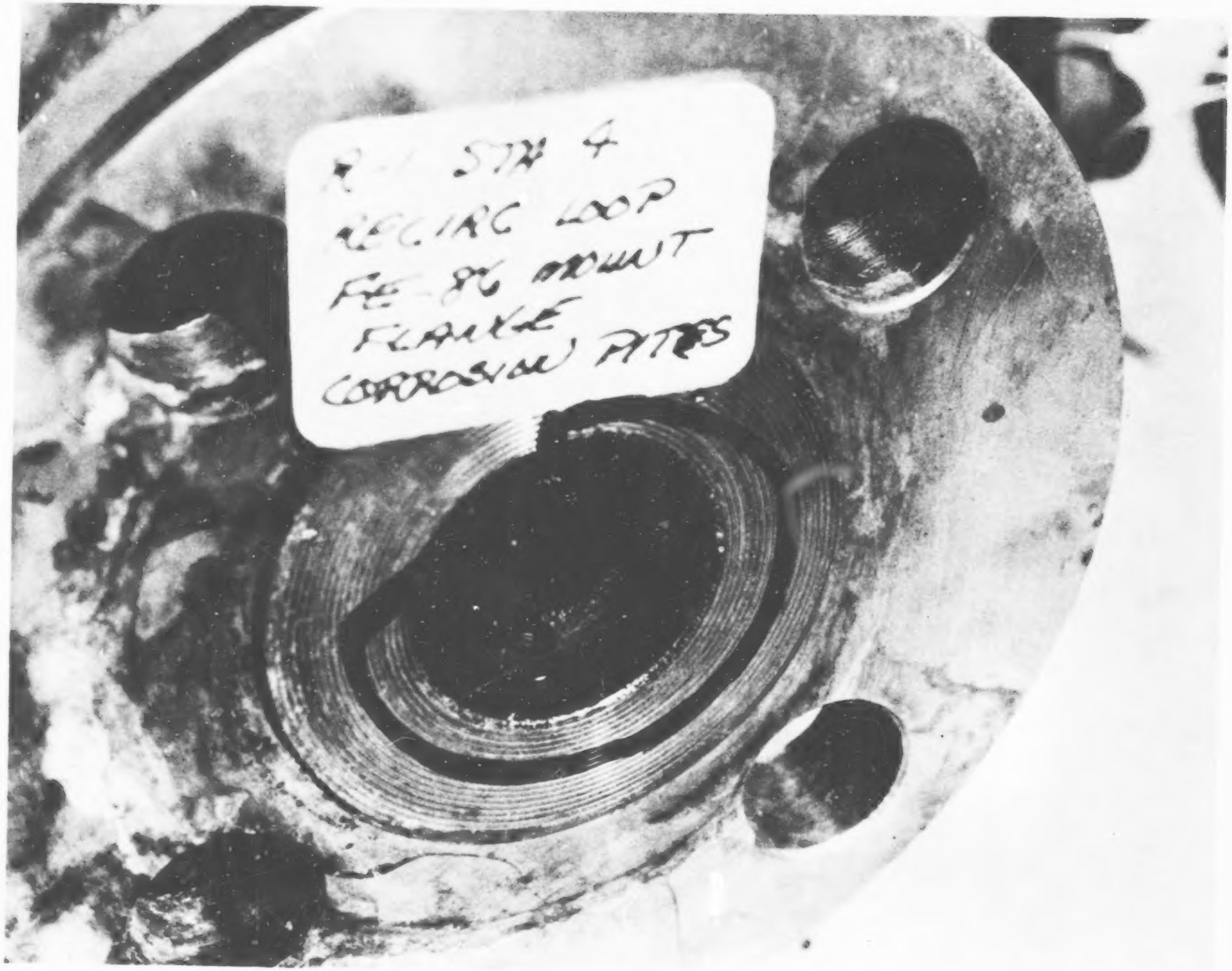

R-1 STA 4
RECIRC LOOP
FE-86 MOUNT
FLANGE
CORROSION PITTES

Figure 38. Flange from Recirculation Loop for Cell 4 of Primary Reactor R-1.

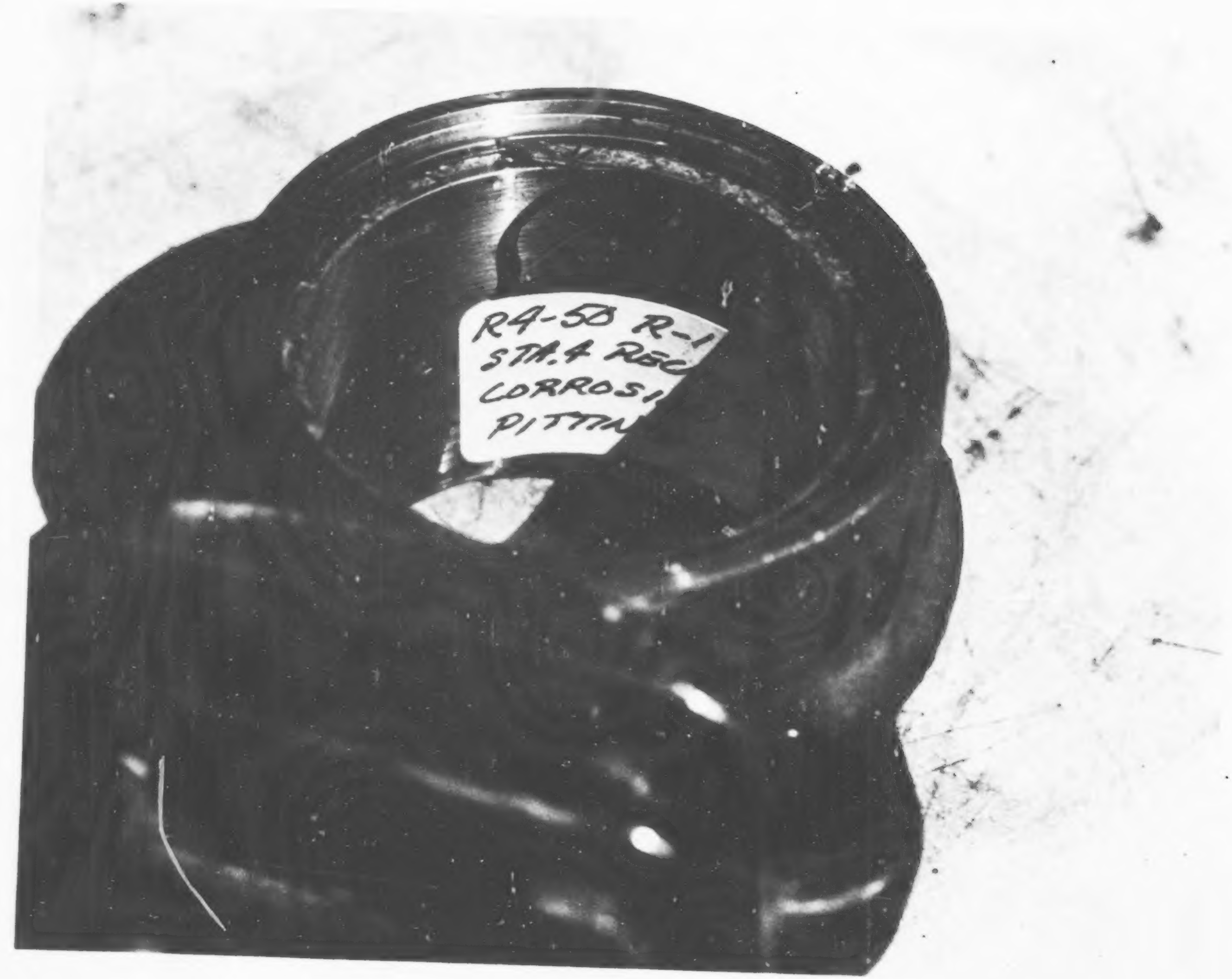

R4-50 R-1
STM.4 REC
CORROSI
PITTIN

Figure 39. Valve Body from Recirculation Loop for Cell 4 of Primary Reactor R-1.

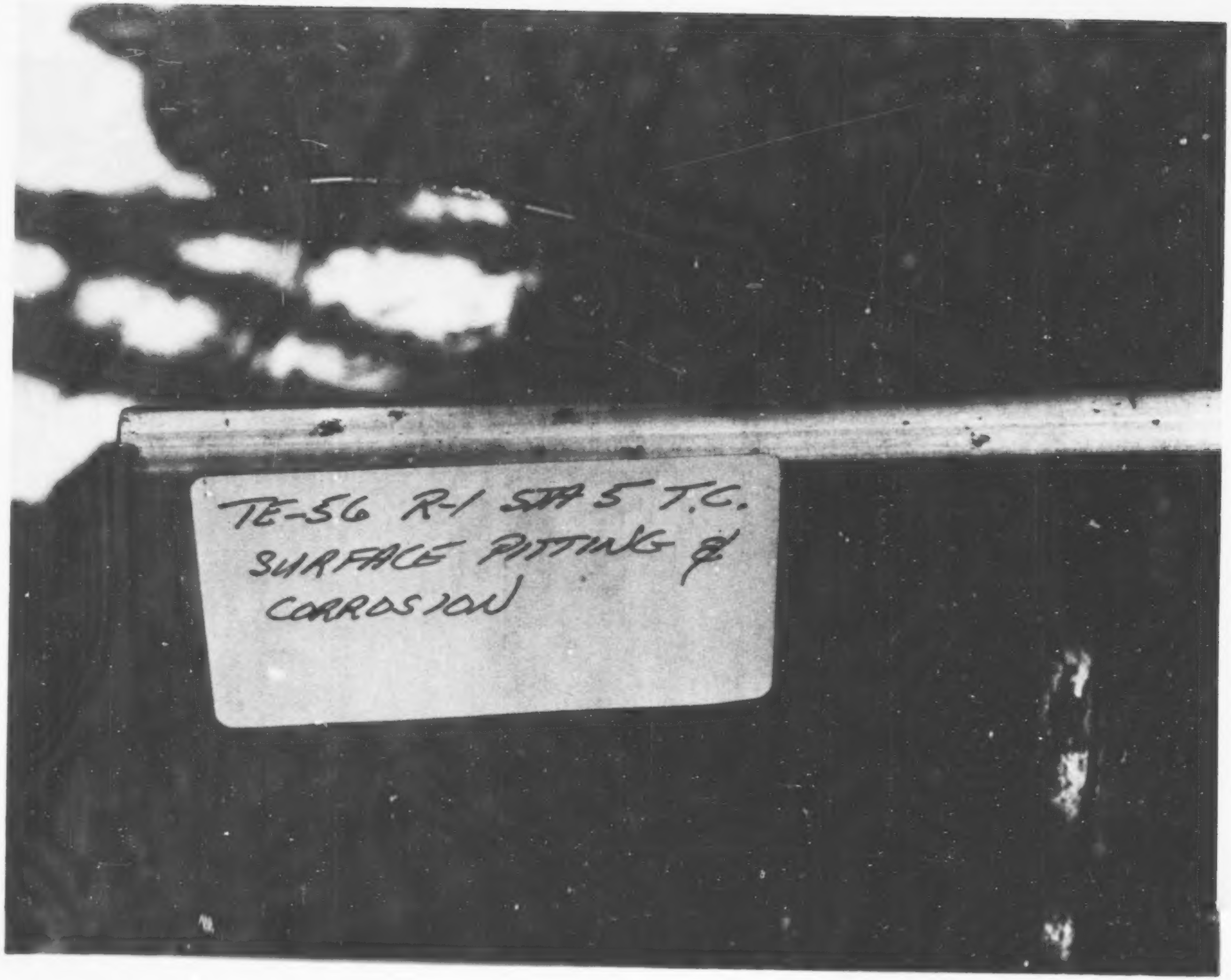

TE-56 R-1 STA 5 T.C.
SURFACE PITTING &
CORROSION

Figure 40. Thermocouple Probe from Cell 5 of Primary Reactor R-1.

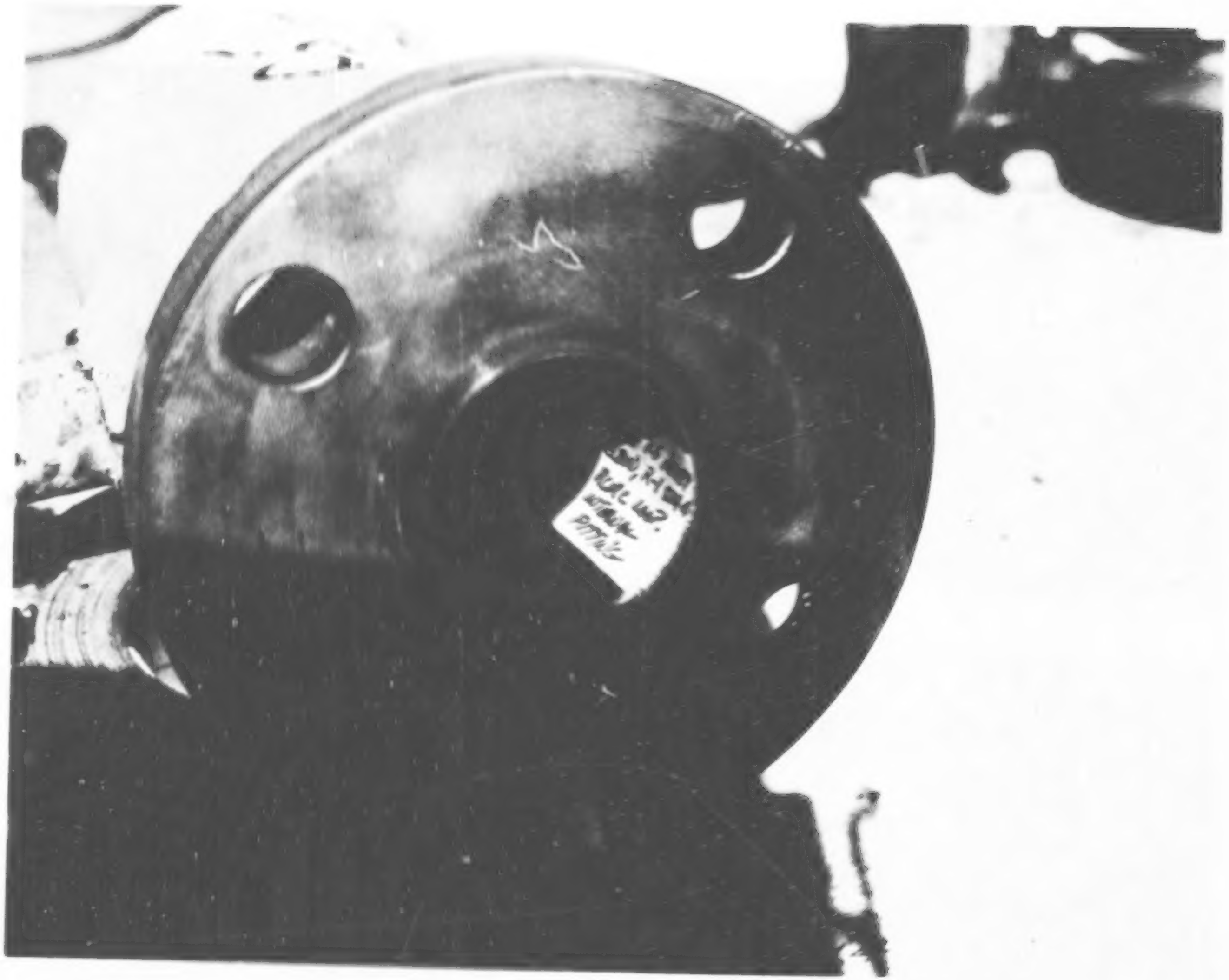


Figure 41. Inlet to Recirculation Pump P-5.

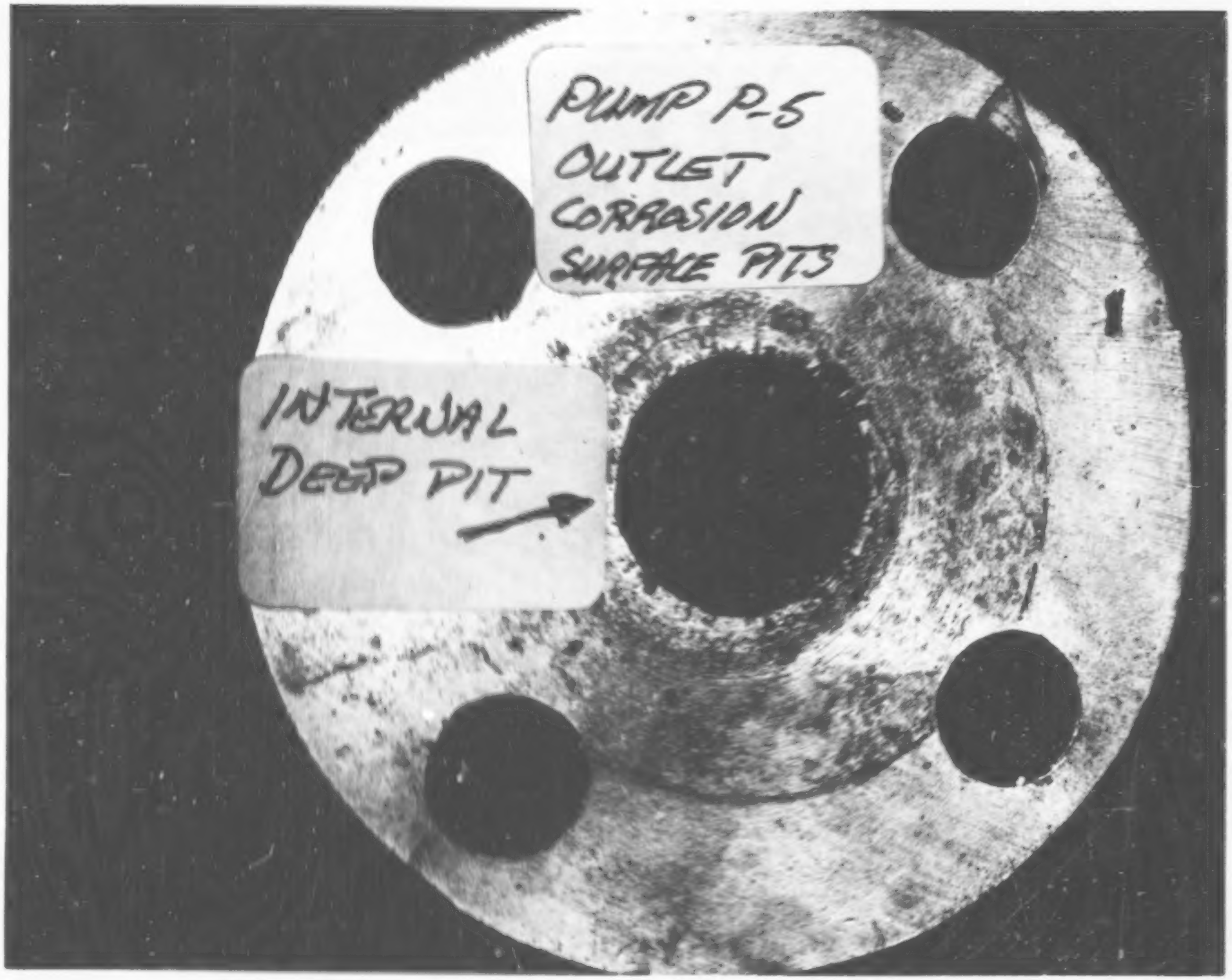

PUMP P-5
OUTLET
CORROSION
SURFACE PITS
INTERNAL
DEEP PIT

Figure 42. Outlet of Recirculation Pump P-5.

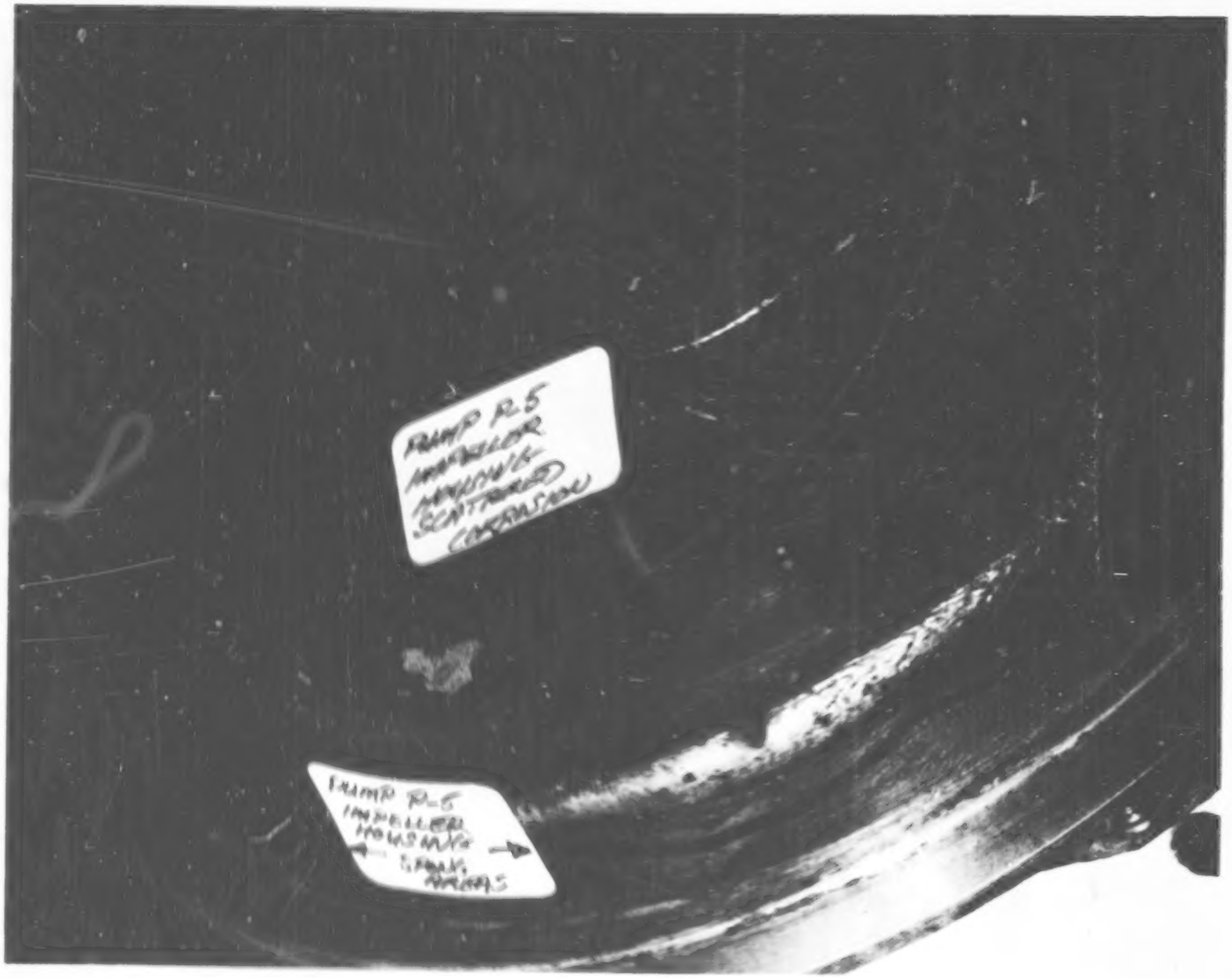

PUMP P-5
IMPELLER
HOUSING
SCATTERED
CORROSION
PUMP P-5
IMPELLER
HOUSING
← SPALL →
AREAS

Figure 43. Pump Housing from Recirculation Pump P-5.

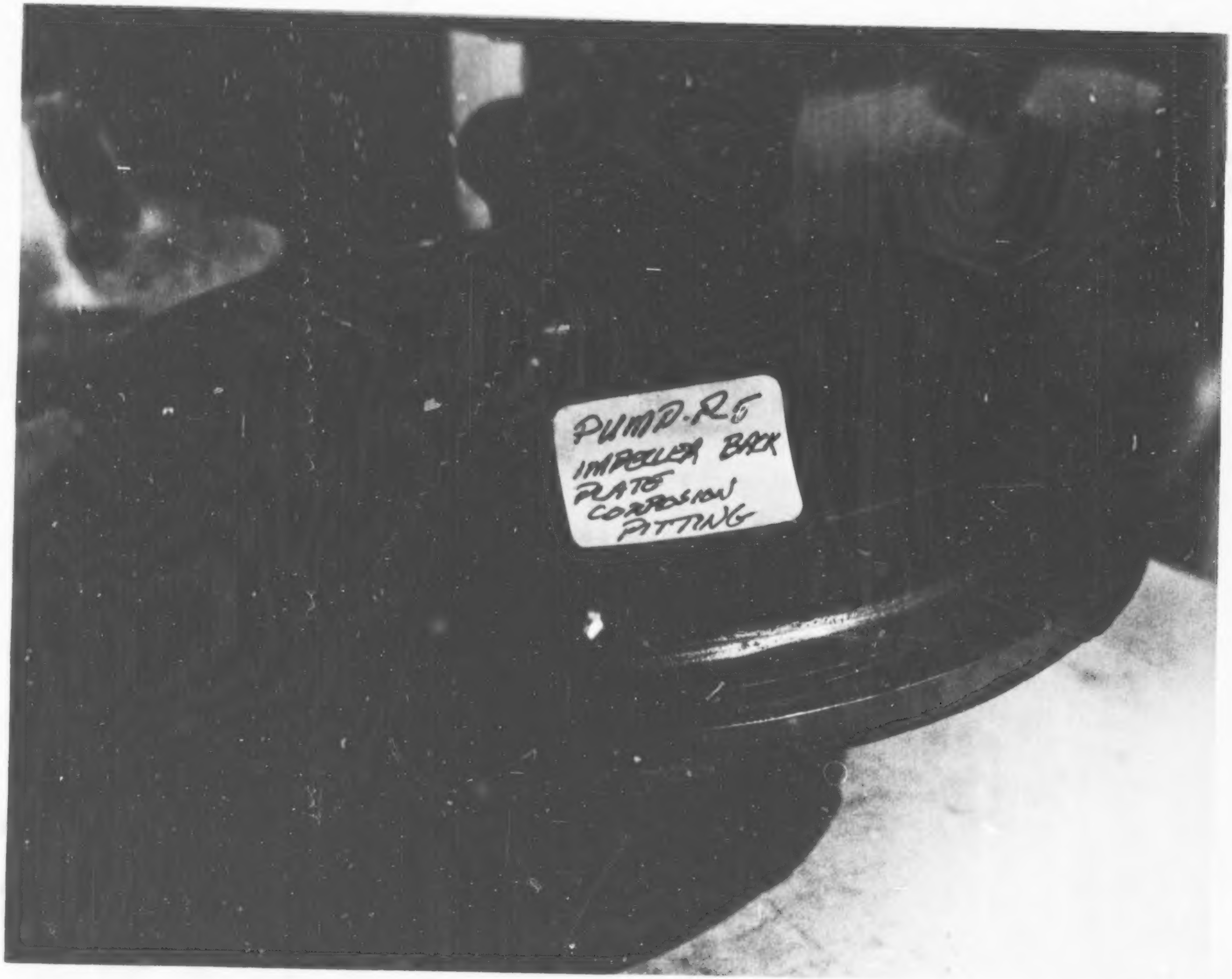

PUMP-P5
IMPELLER BACK
PLATE
CORROSION
PITTING

Figure 44. Impeller Back Plate from Recirculation Pump P-5 - Side View.

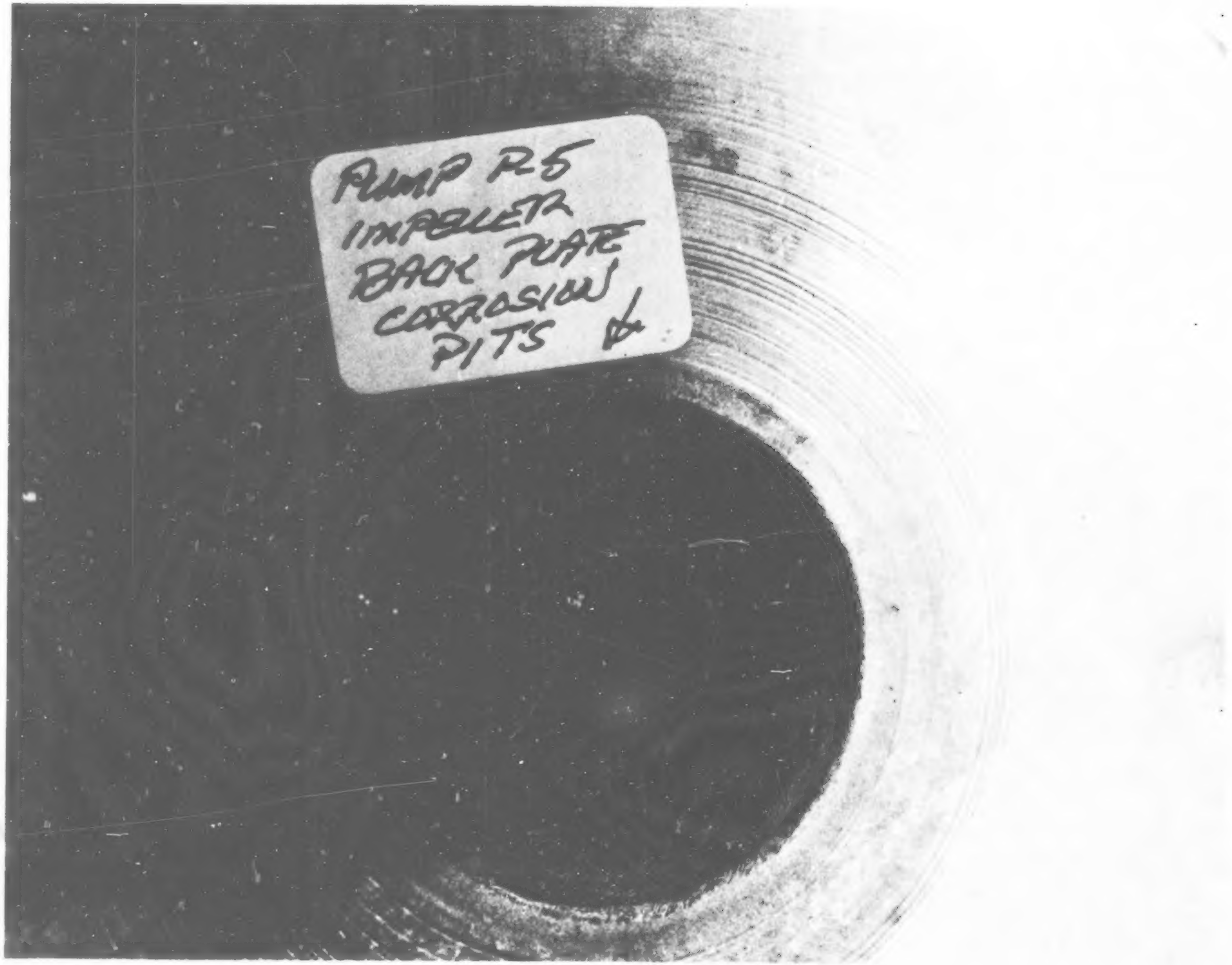

PUMP P-5
IMPELLER
BACK PLATE
CORROSION
PITS

Figure 45. Impeller Back Plate from Recirculation Pump P-5 - End View.

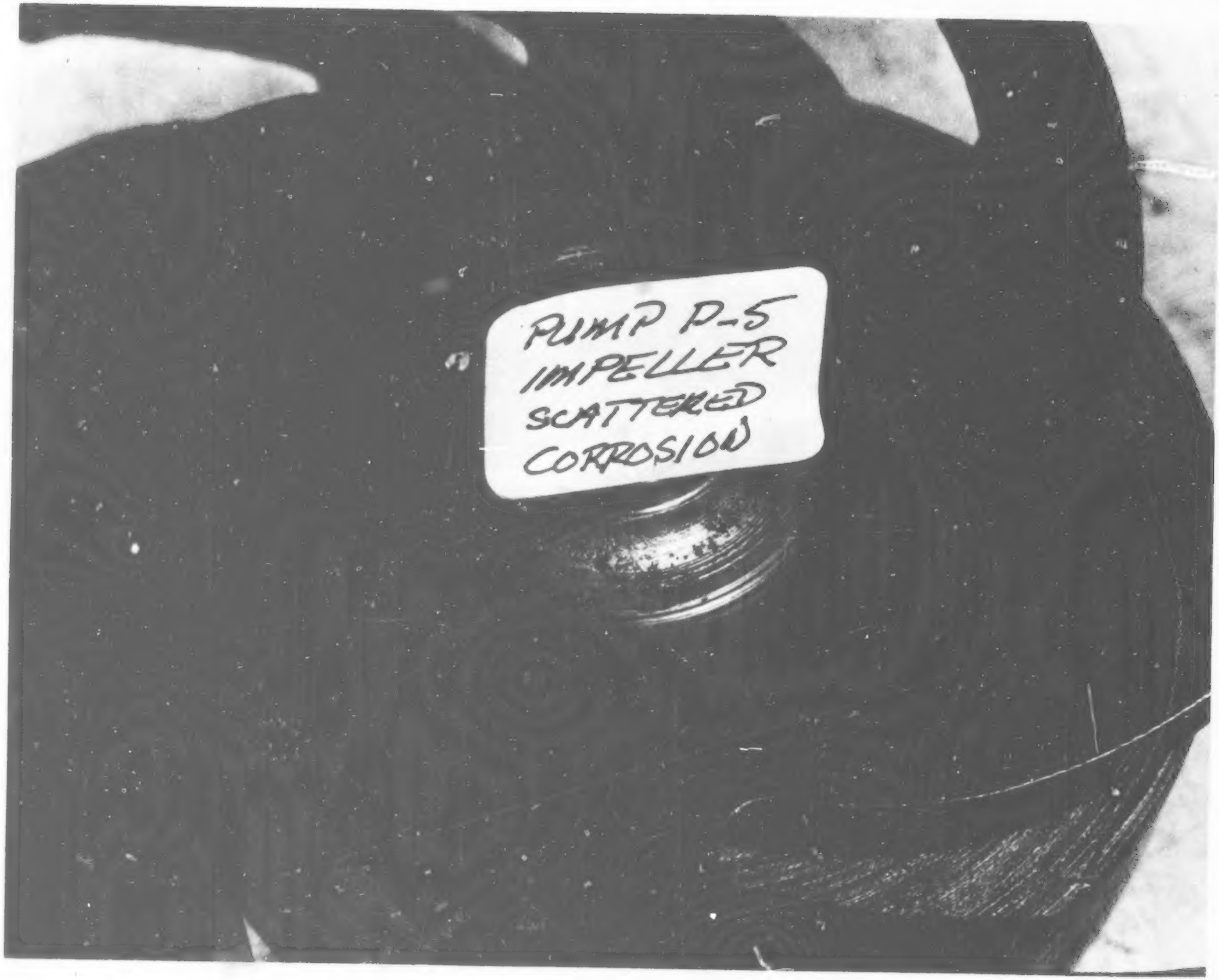

PUMP P-5
IMPELLER
SCATTERED
CORROSION

Figure 46. Impeller from Recirculation Pump P-5. Front View.

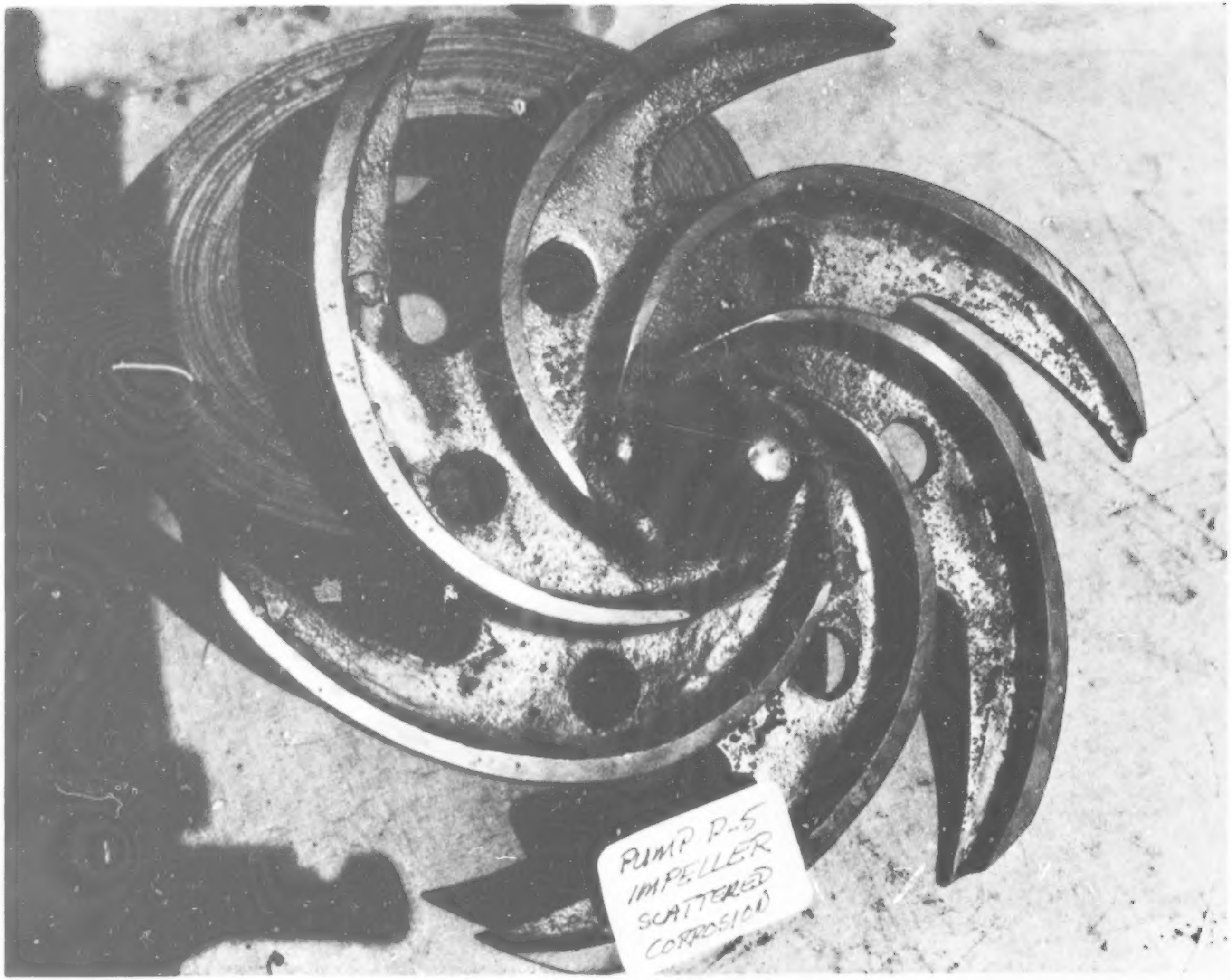

PUMP P-5
IMPELLER
SCATTERED
CORROSION

Figure 47. Impeller from Recirculation Pump P-5 - Back View.

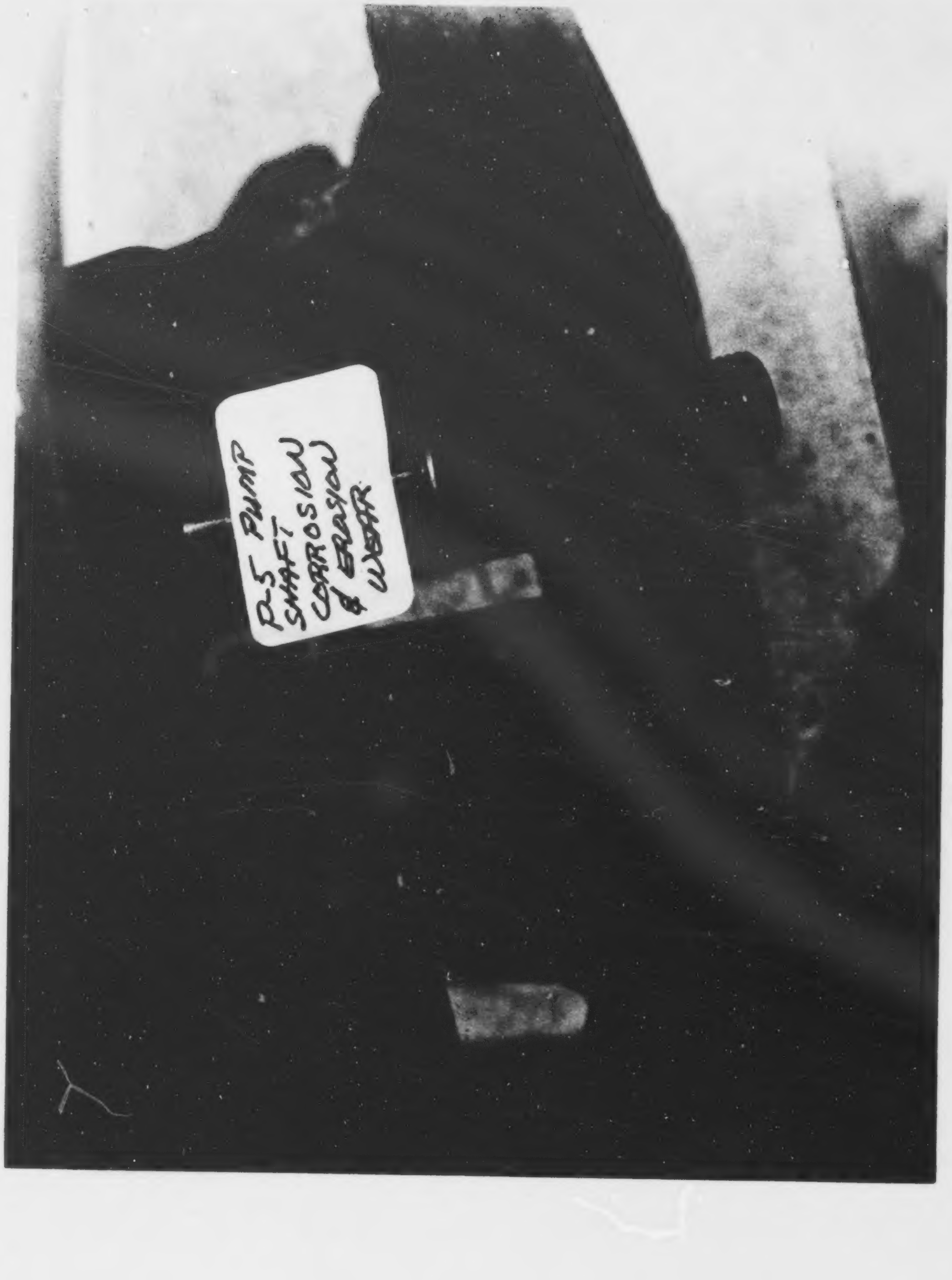

P-5 PUMP
SHAFT
CORROSION
& EROSION
WEAR

Figure 48. Drive Shaft from Recirculation Pump P-5.

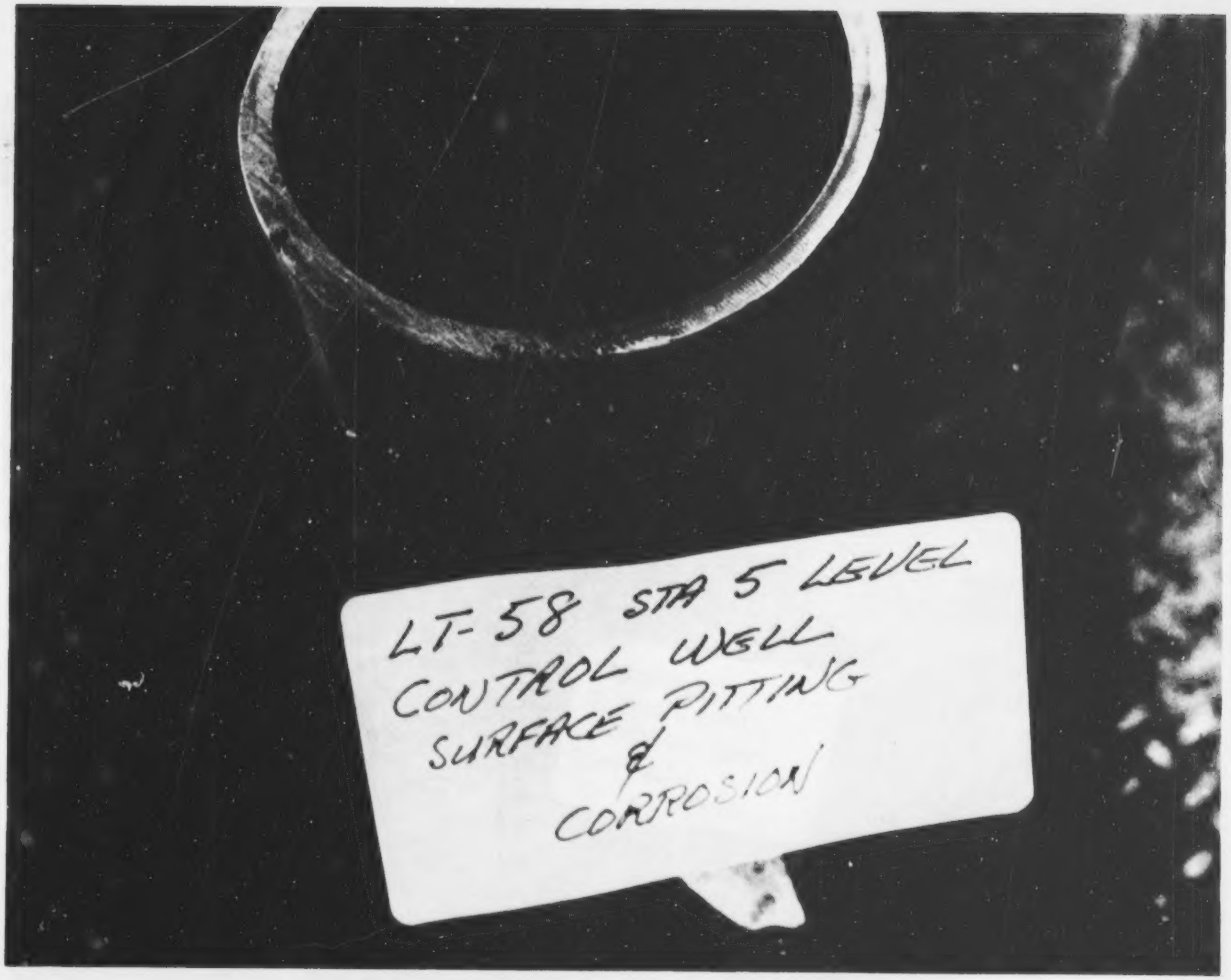

LT-58 STA 5 LEVEL
CONTROL WELL
SURFACE PITTING
&
CORROSION

Figure 49. Float Well for Level Monitor Gauge LT-58 from Cell 5 of Primary Reactor R-1.

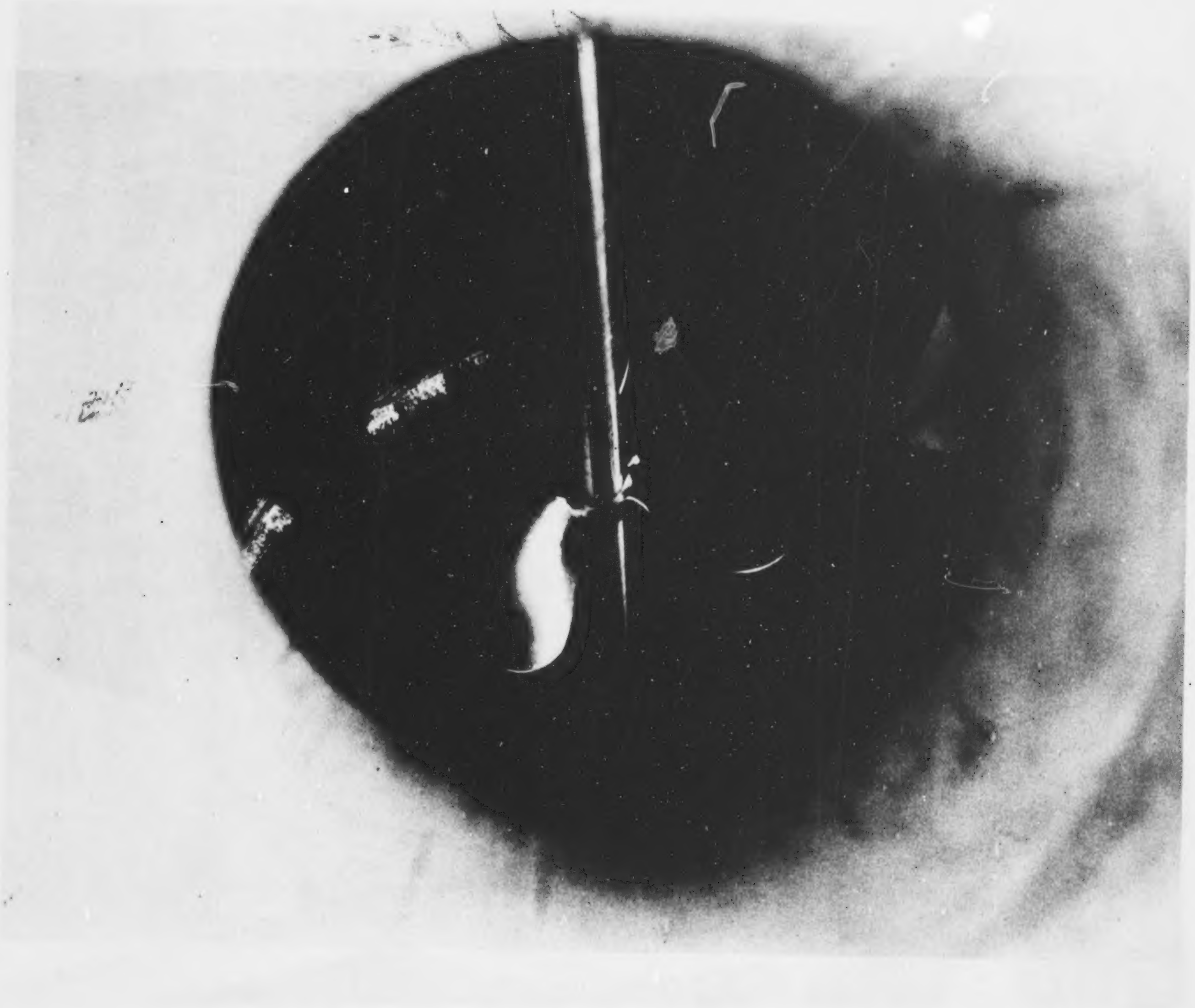


Figure 50. Interior View of Secondary Reactor R-1.

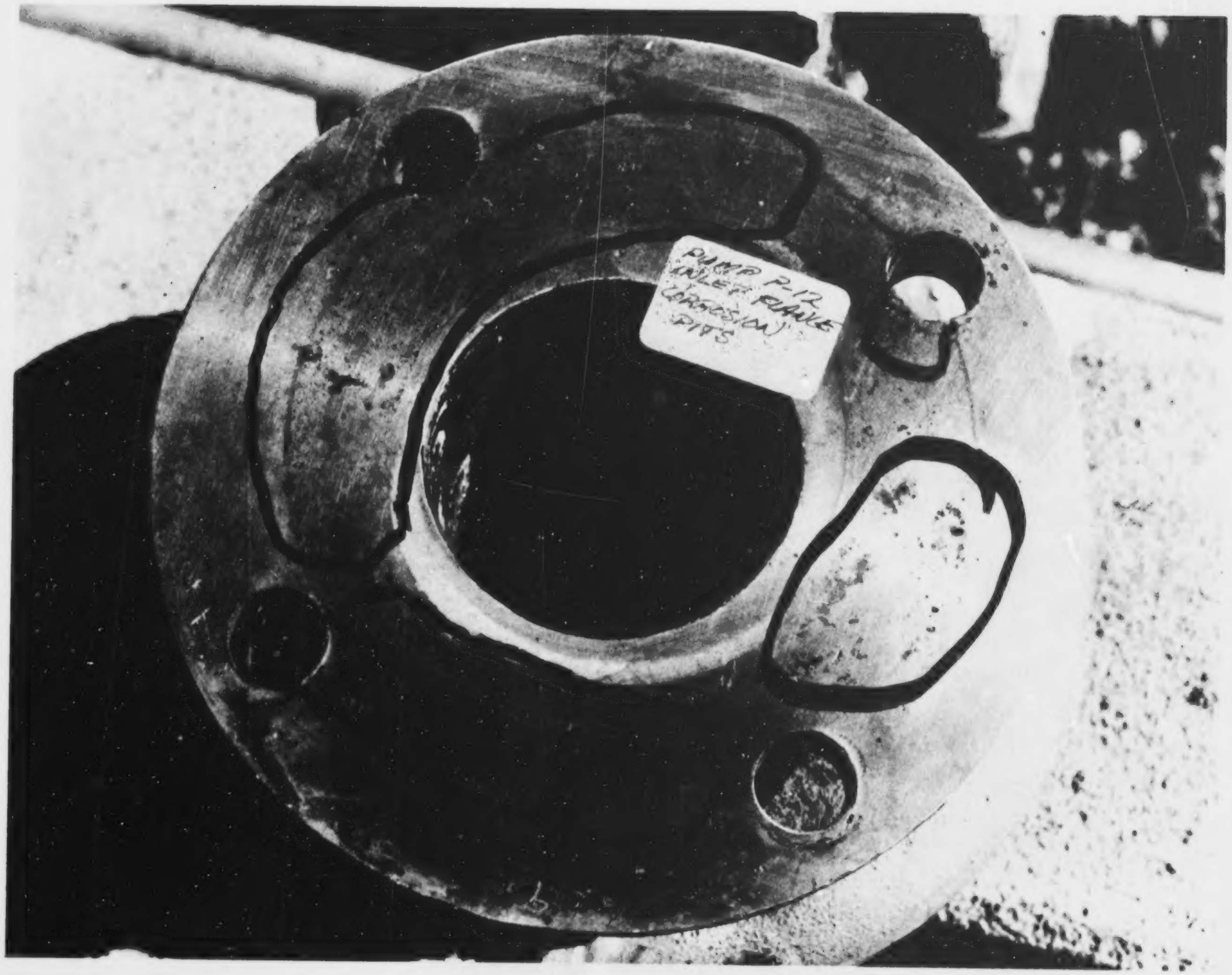

PUMP P-12
INLET FLANGE
CORROSION
PITS

Figure 51. Inlet Flange Face of Leach Solution Circulation Pump P-12.

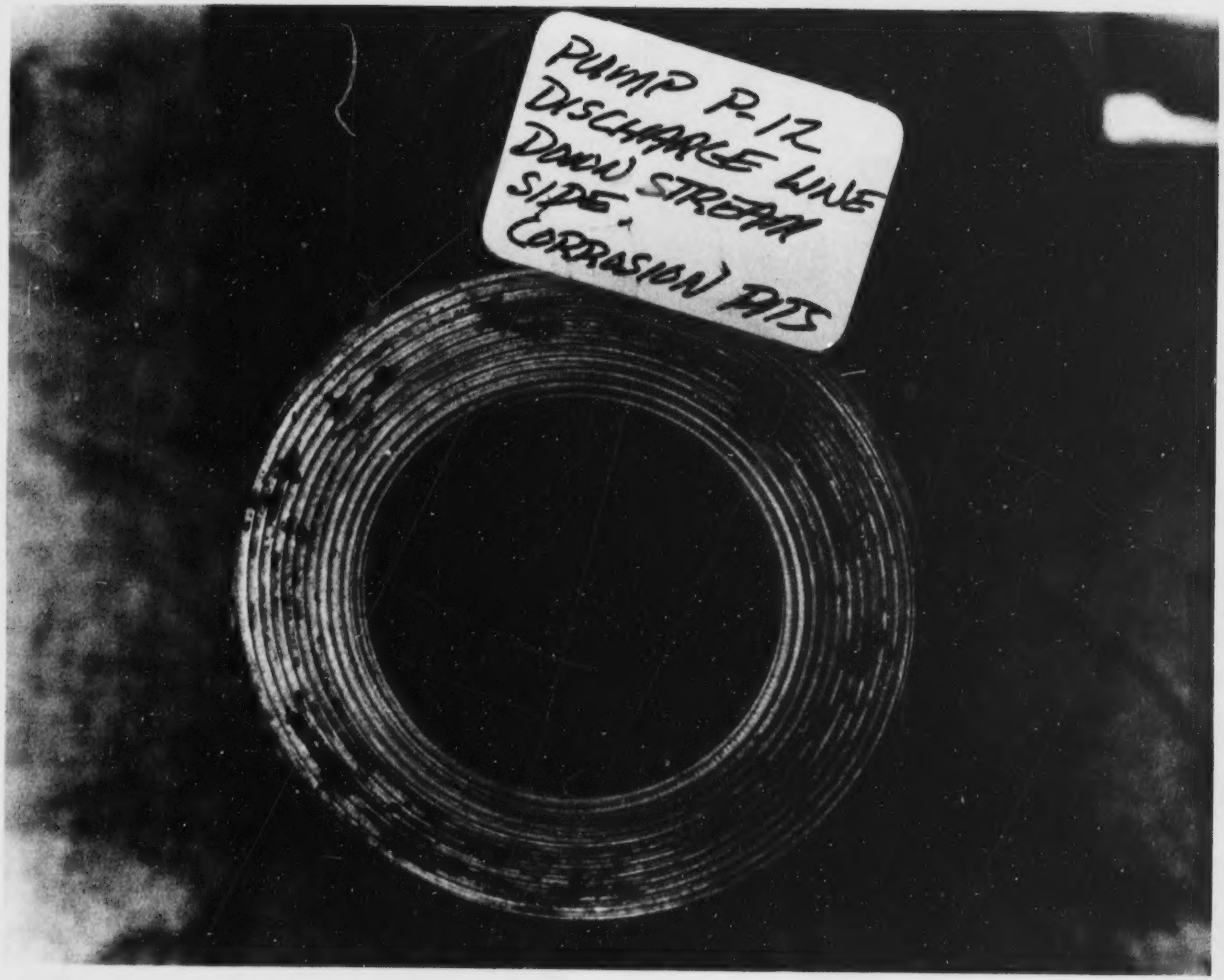

PUMP P-12
DISCHARGE LINE
DOWN STREAM
SIDE.
CORRASION PITS

Figure 52. Outlet Flange Face on Leach Solution Circulation Pump P-12.

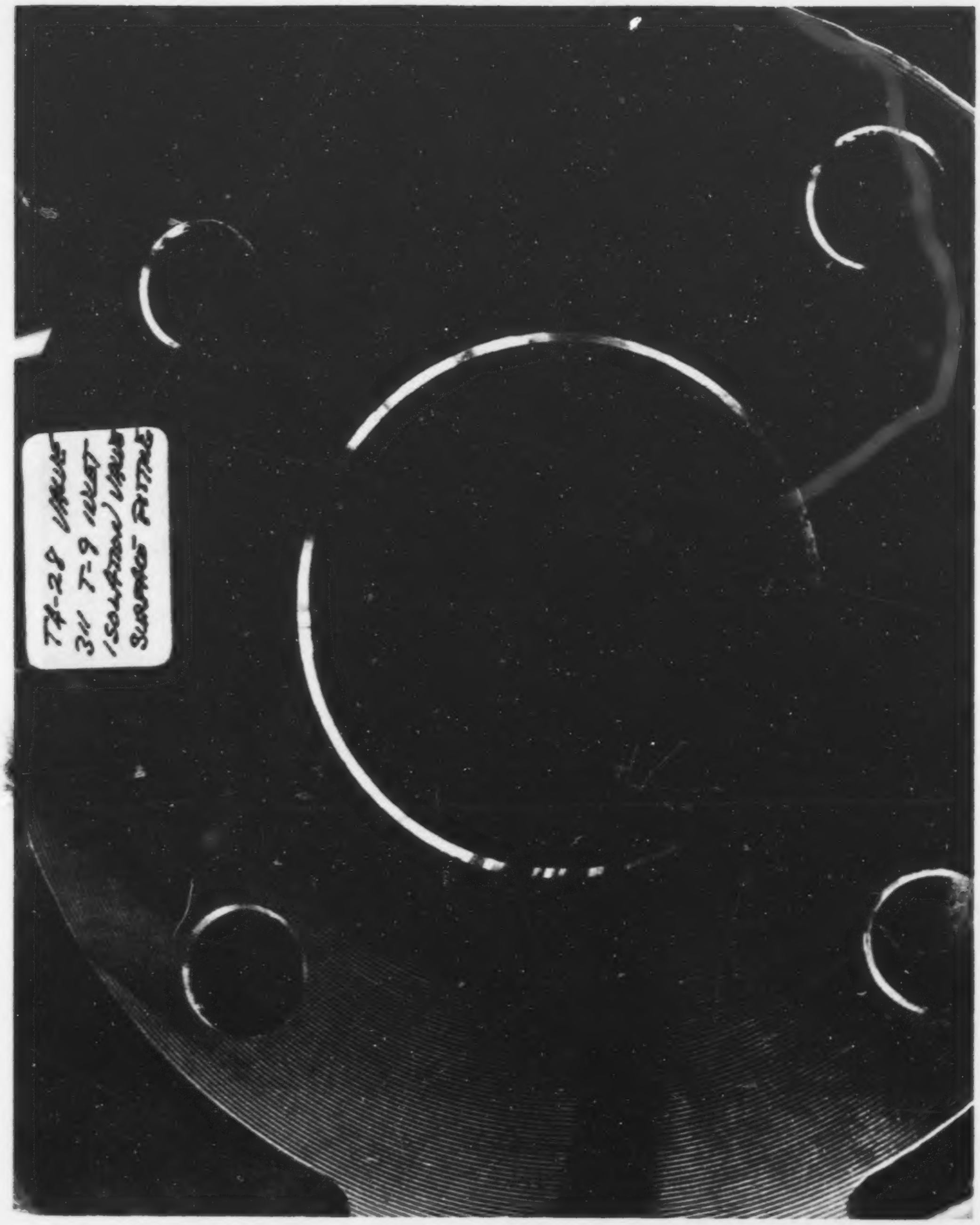

T4-28 VALVE
3" T-9 INLET
ISOLATION VALVE
SURFACE FITTING

Figure 53. Flange Face in Inlet Isolation Valve T4-28 to Tank T-9.

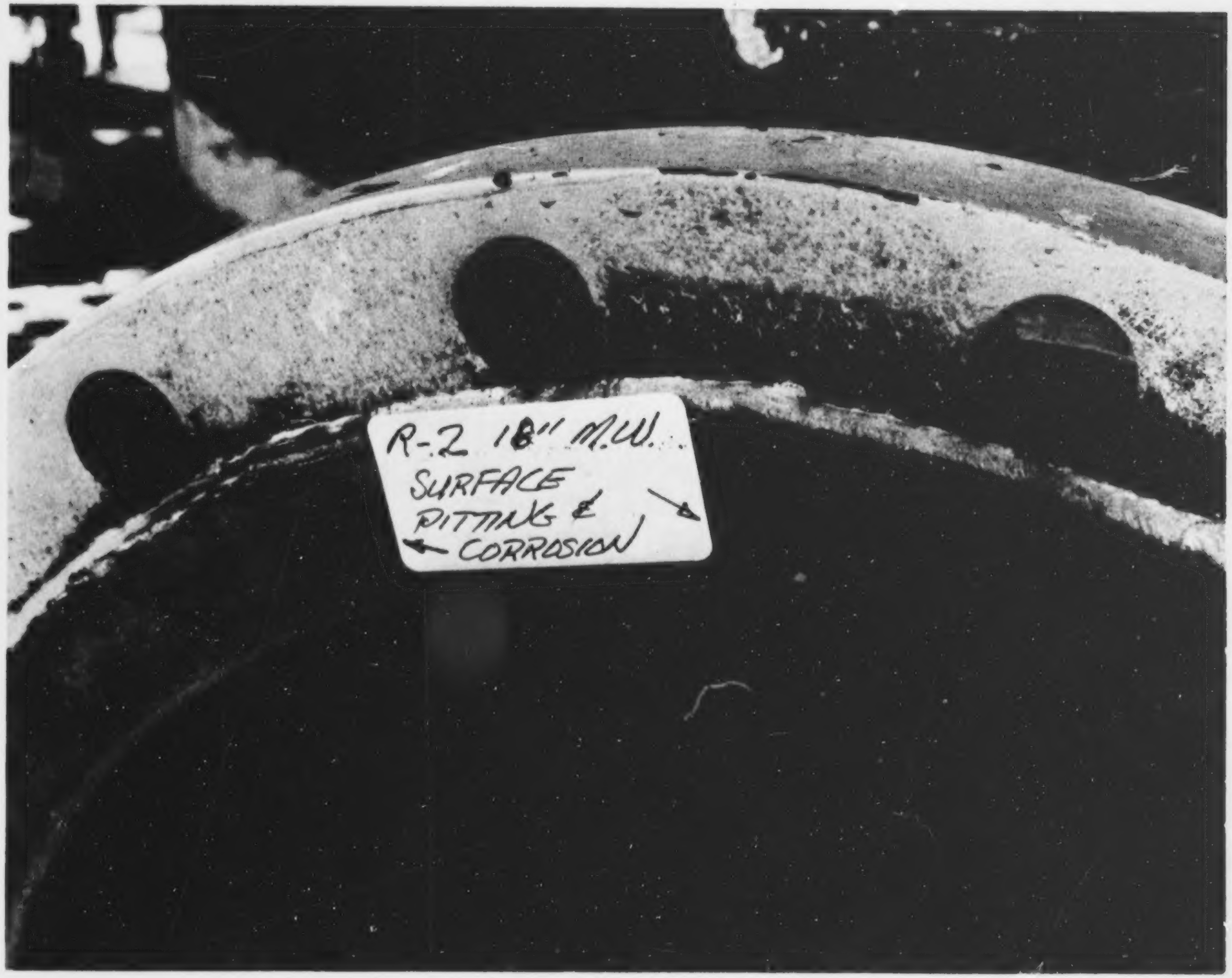

R-2 18" M.W.
SURFACE
PITTING &
CORROSION

Figure 54. Flange Face on Reactor R-2 Manway.

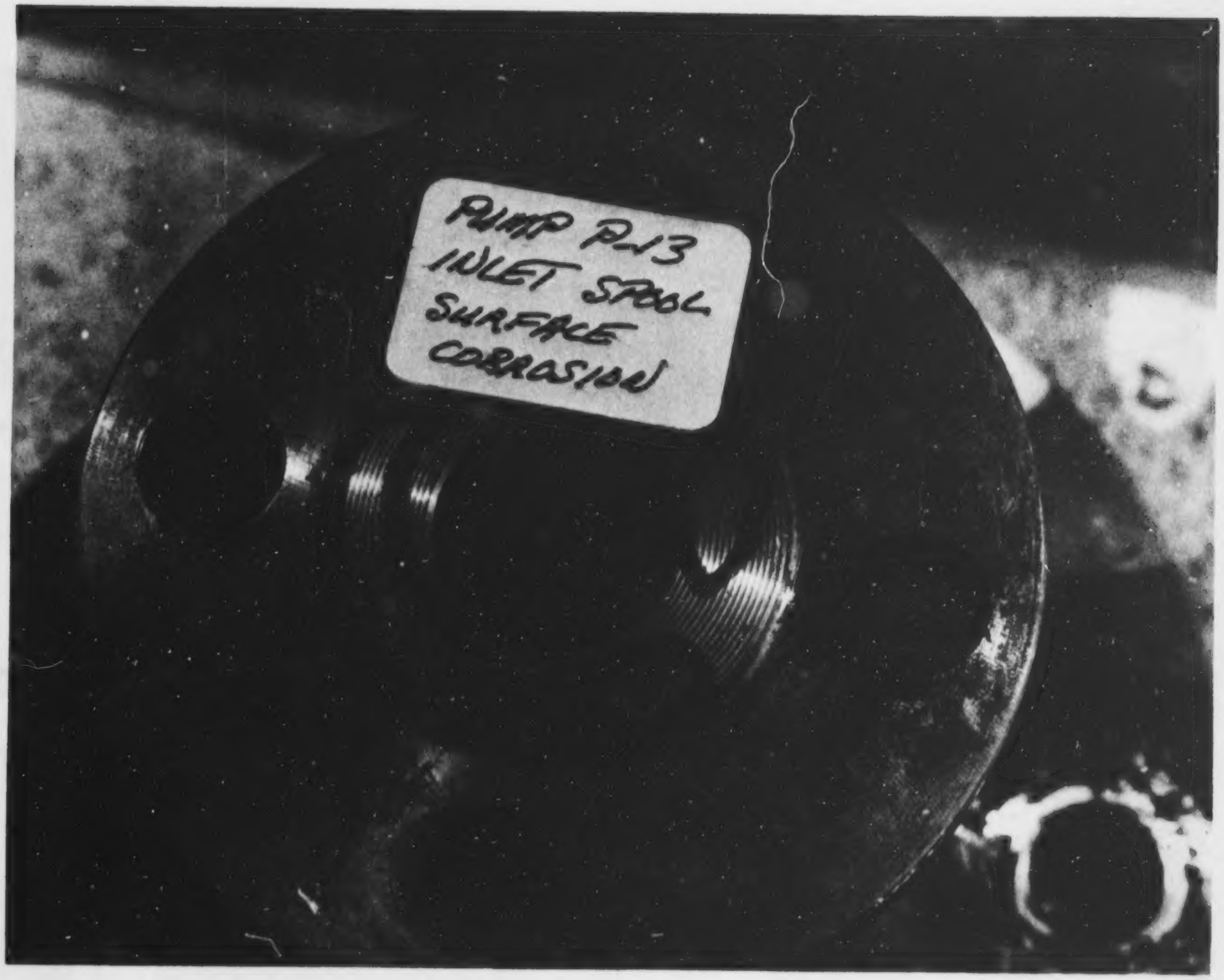

PUMP P-13
INLET SPOOL
SURFACE
CORROSION

Figure 55. Flange Face on Inlet Spool to Leach Solution Pump P-13.

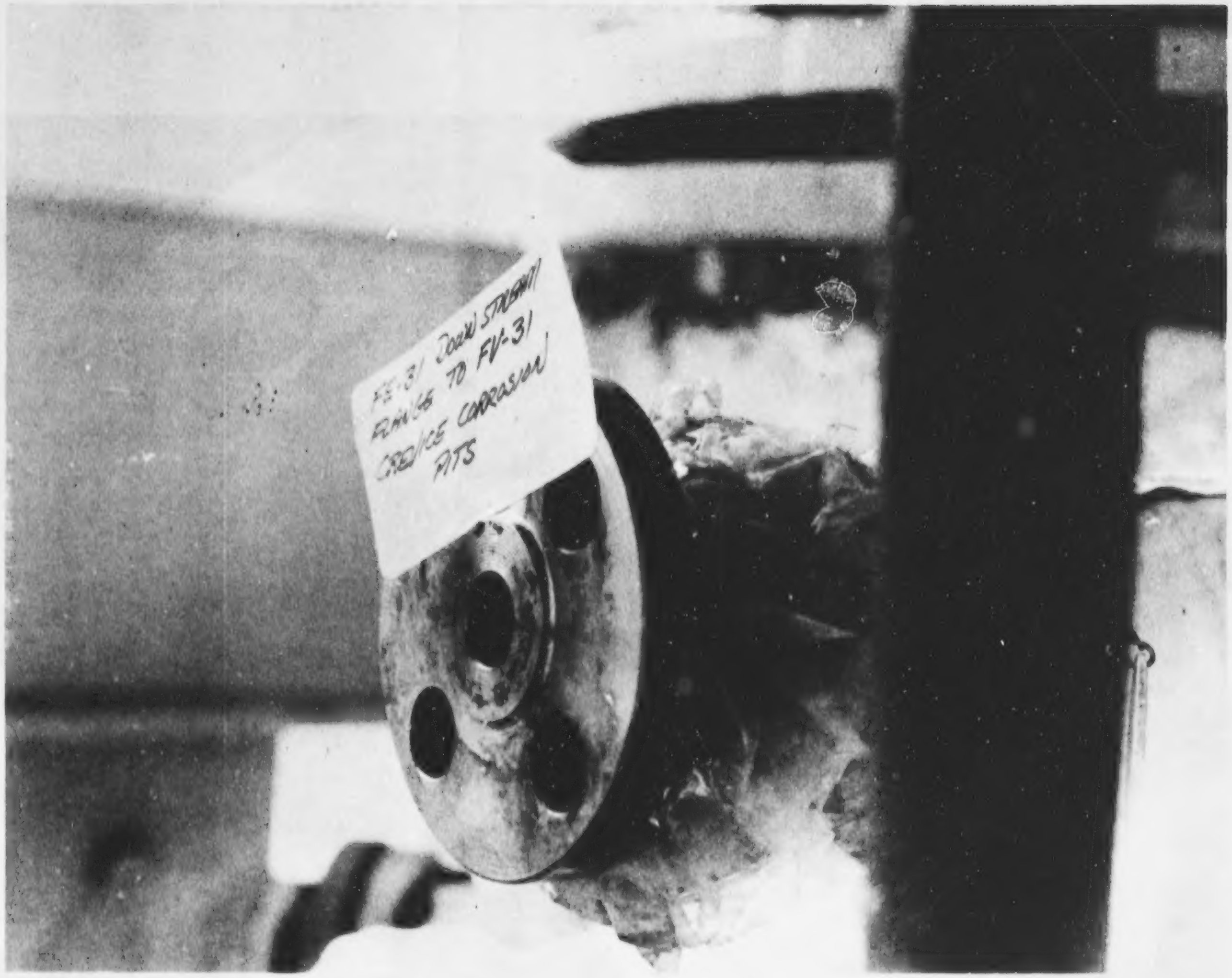

FE-31 DOWNSTREAM
FLANGE TO FV-31
CREVICE CORROSION
PITS

Figure 56. Flange Face from Leach Solution Feed Line to Foam Scrubber T-3.

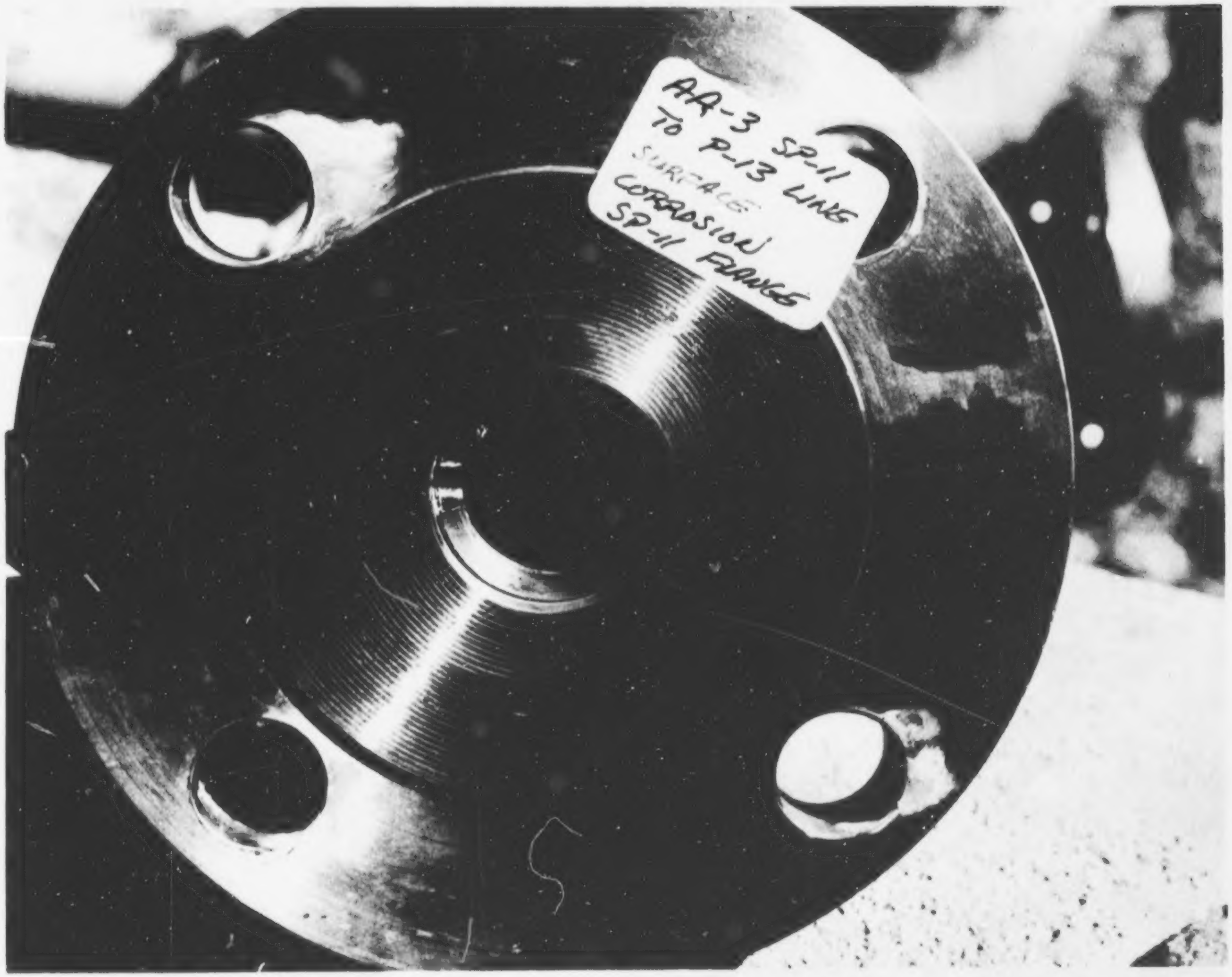

AA-3 SP-11
TO P-13 LINE
SURFACE
CORROSION
SP-11 FLANGE

Figure 57. Flange Face from Leach Solution Feed Line to Pump P-13.

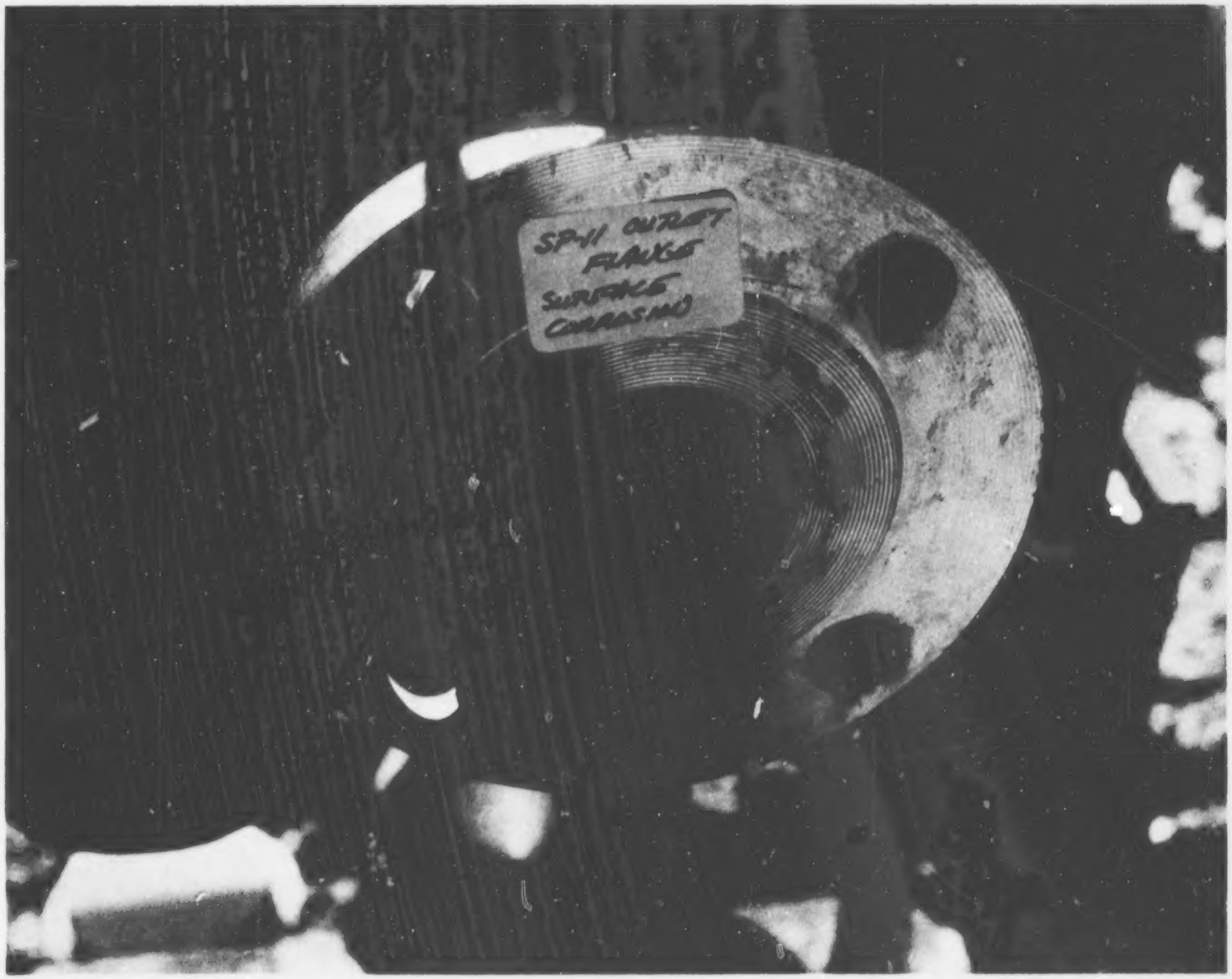

SP-11 OUTLET
FLANGE
SURFACE
CORROSION

Figure 58. Flange Face on Basket Strainer SP-11 Outlet Port.

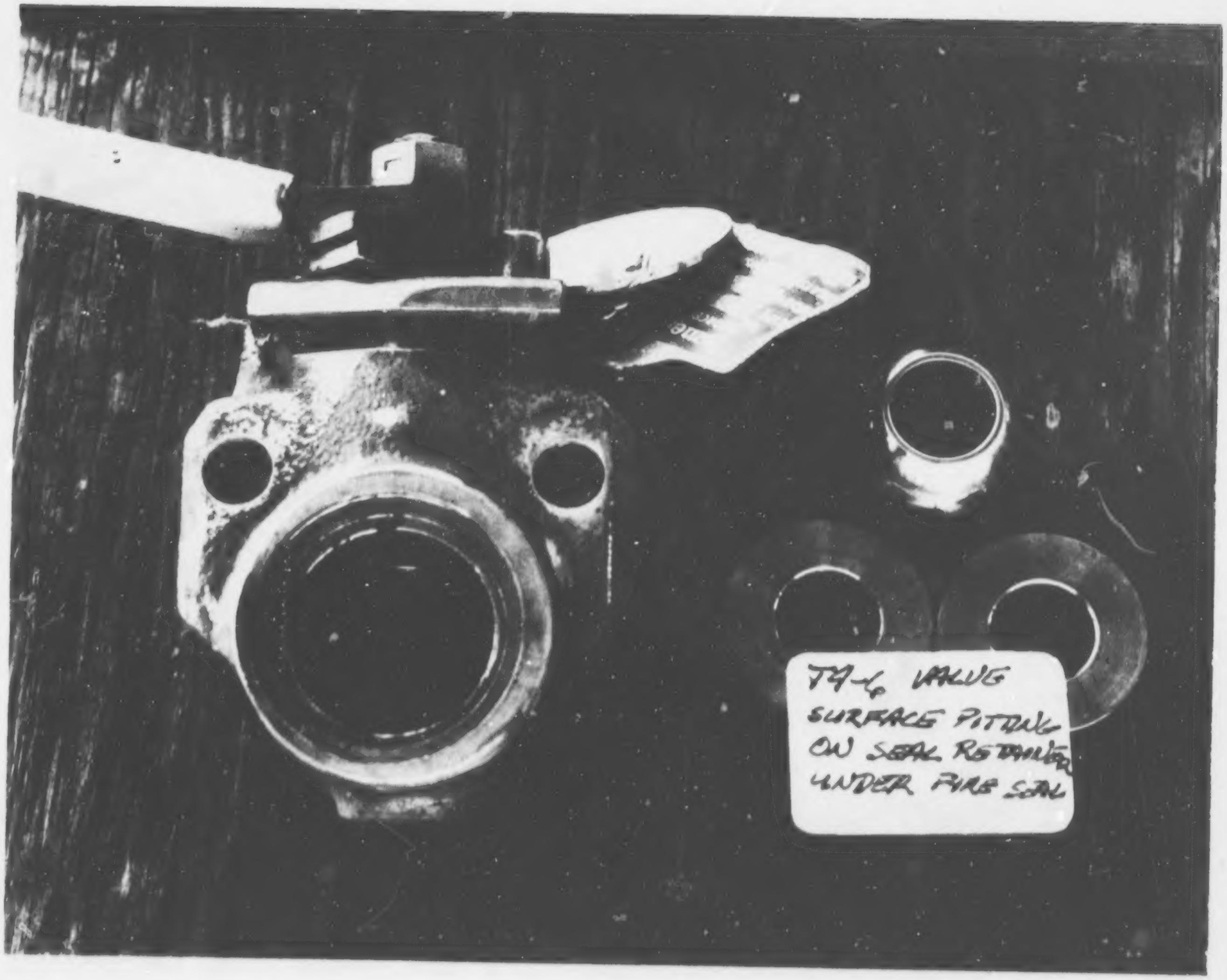

T4-6 VALVE
SURFACE PITTING
ON SEAL RETAINER
UNDER FIRE SEAL

Figure 59. Disassemble Valve T4-6 from Filter Cake Wash Line to Tank T-9.

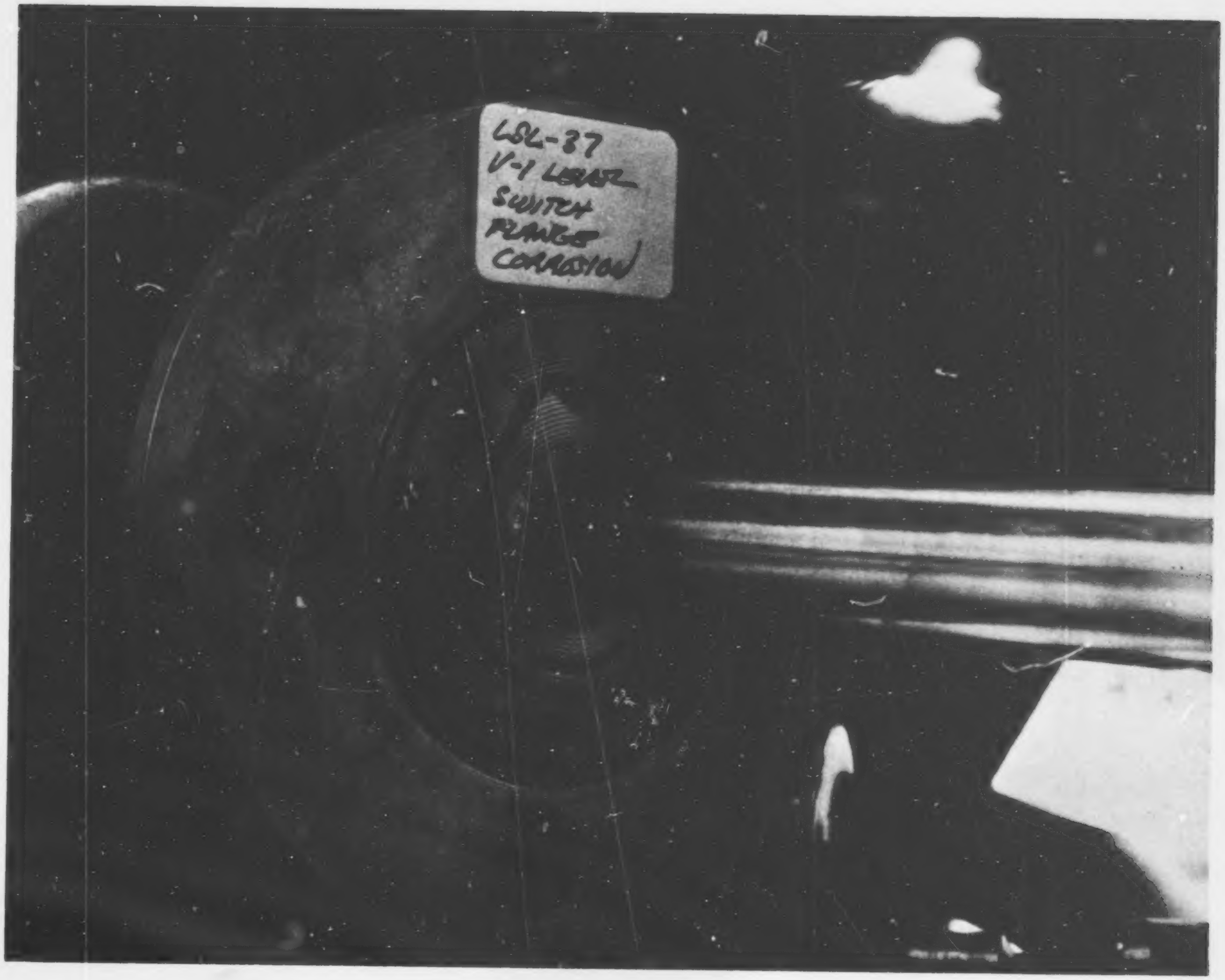

LSL-37
V-1 LEVER
SWITCH
FLANGE
CORROSION

Figure 60. Level Switch LSL-37 from Knockout Drum V-1.

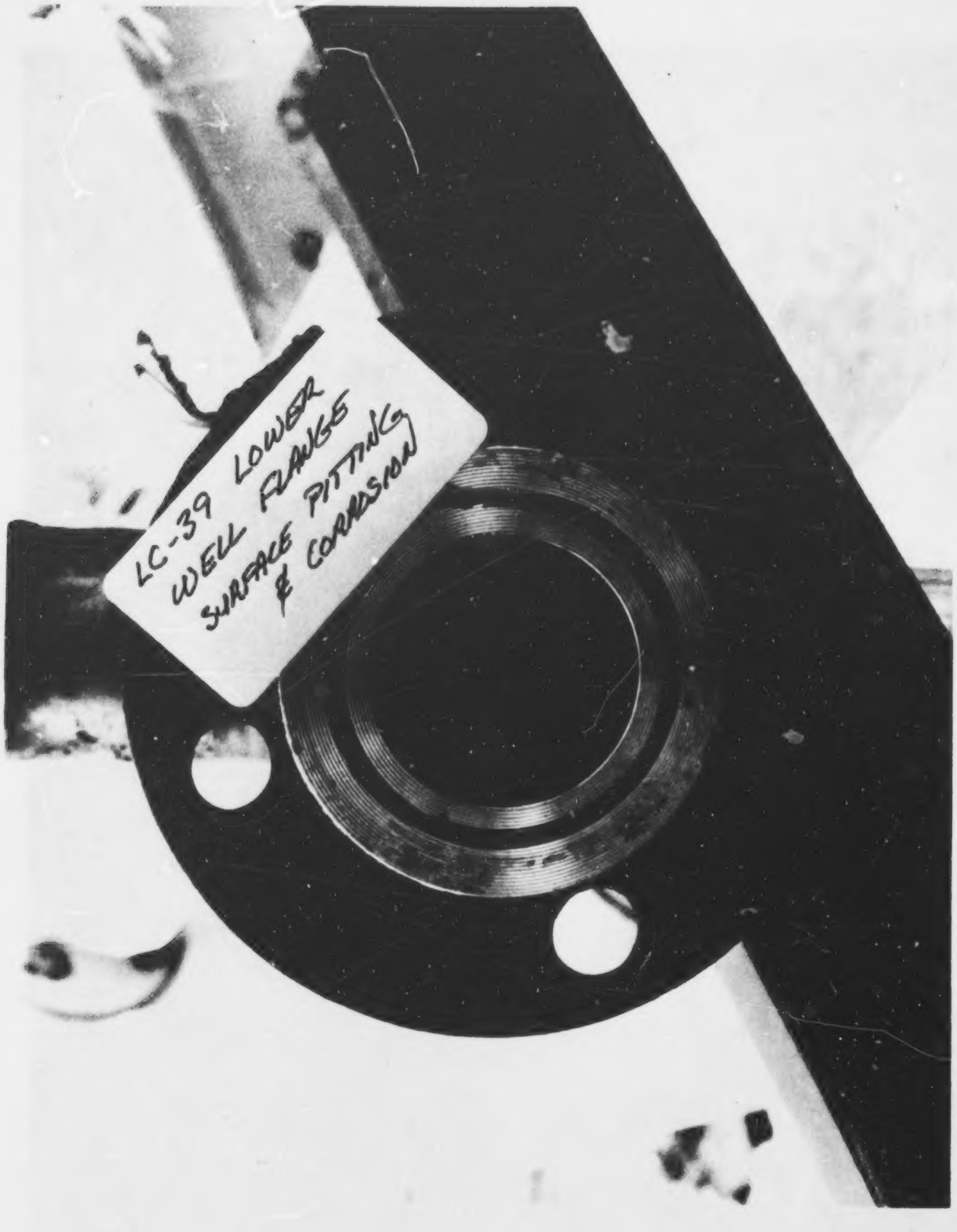

LC-39 LOWER
WELL FLANGE
SURFACE PITTING
& CORROSION

Figure 61. Lower Flange Face on Level Controller LC-39 for Knockout Drum V-1.

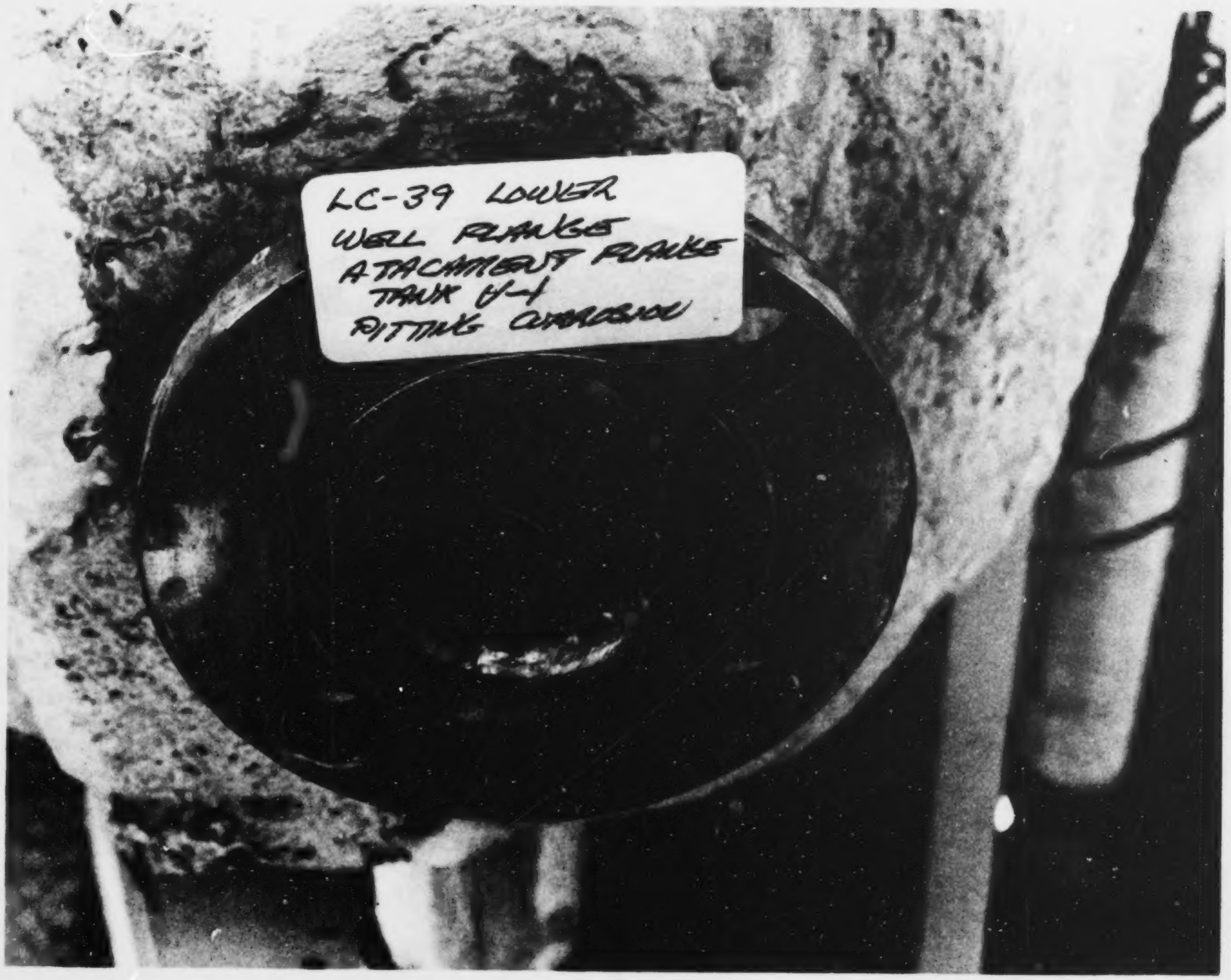

LC-39 LOWER
WELL FLANGE
ATTACHMENT FLANGE
TANK V-1
PITTING CORROSION

Figure 62. Lower Flange Face on Knockout Drum V-1.

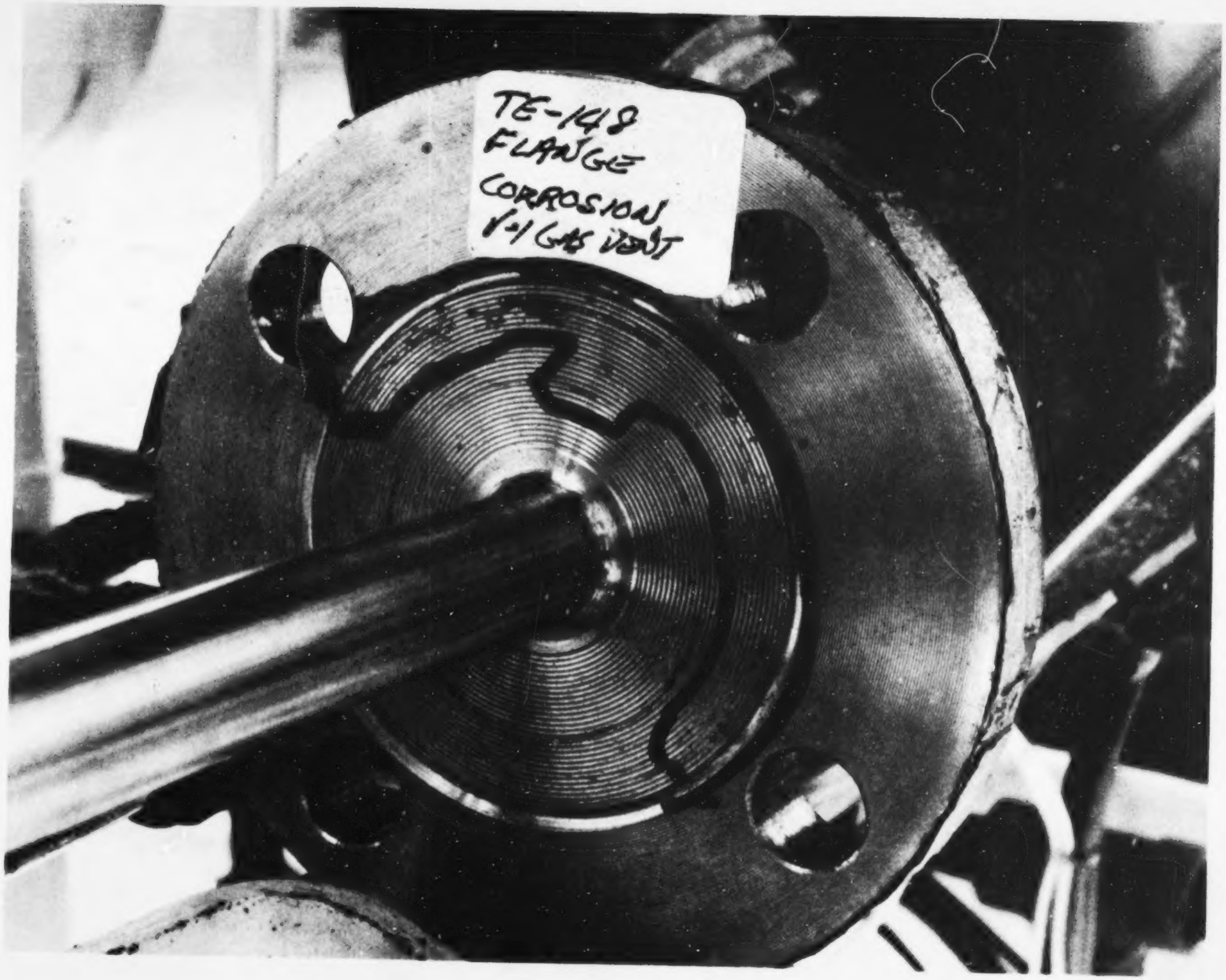

TE-148
FLANGE
CORROSION
V-1 GAS VENT

Figure 63. Thermocouple Probe TE-148 from Knockout Drum V-1.

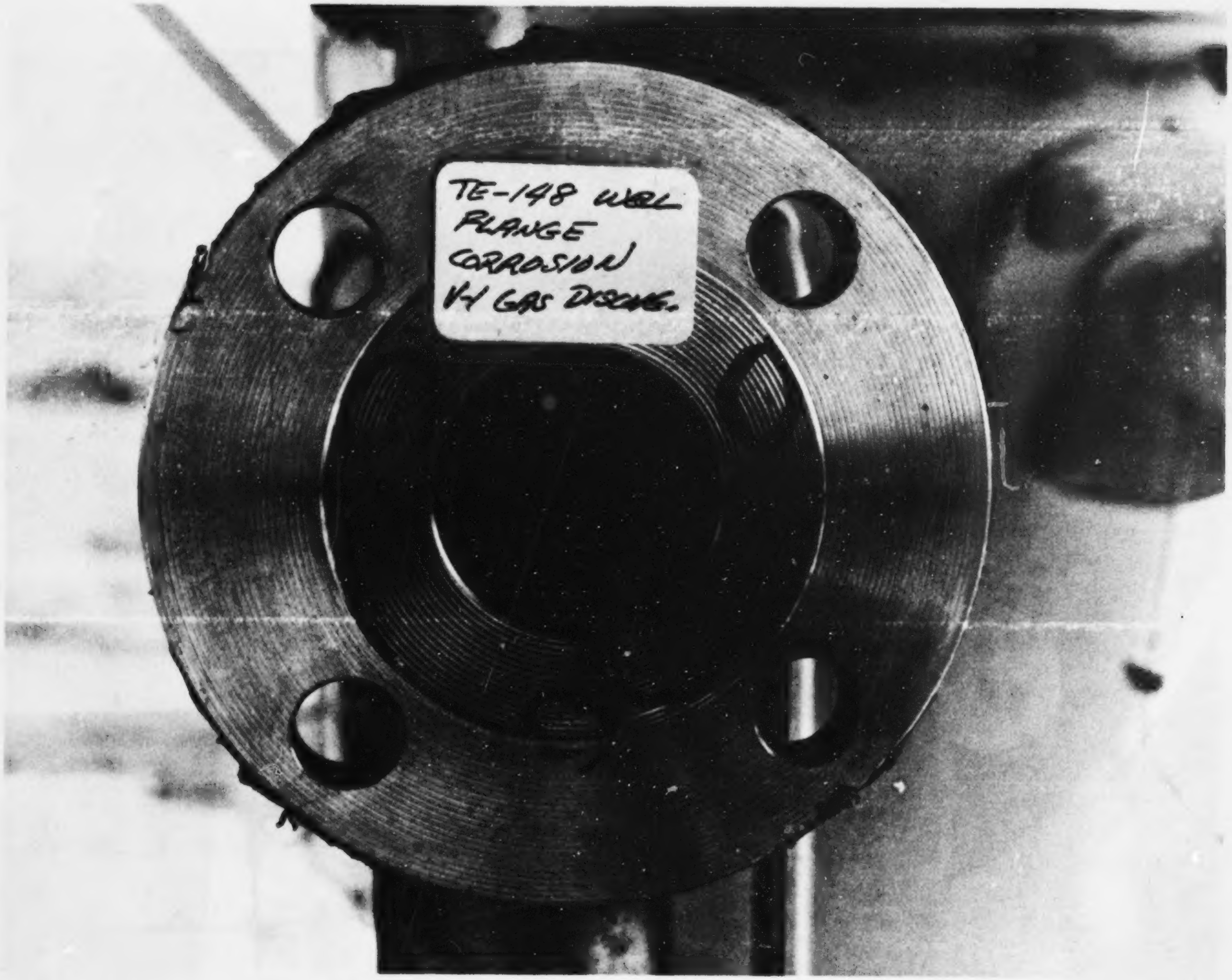

TE-148 WELL
FLANGE
CORROSION
V-1 GAS DISCHG.

Figure 64. Mating Flange to Thermocouple Probe TE-148.

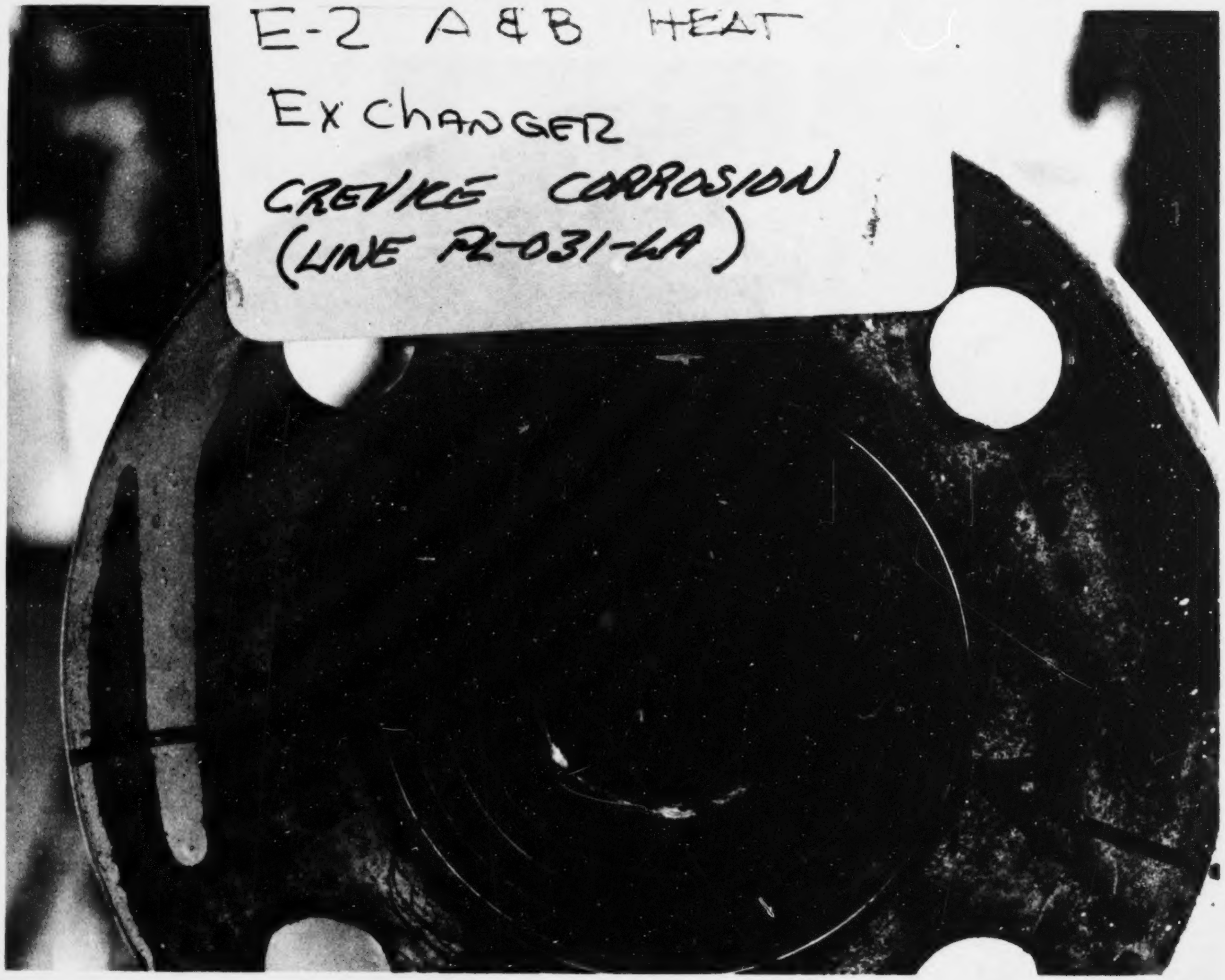

E-2 A&B HEAT
EXCHANGER
CREVICE CORROSION
(LINE P2-031-6A)

Figure 65. Flange Face at Outlet of Heat Exchanger E-2.

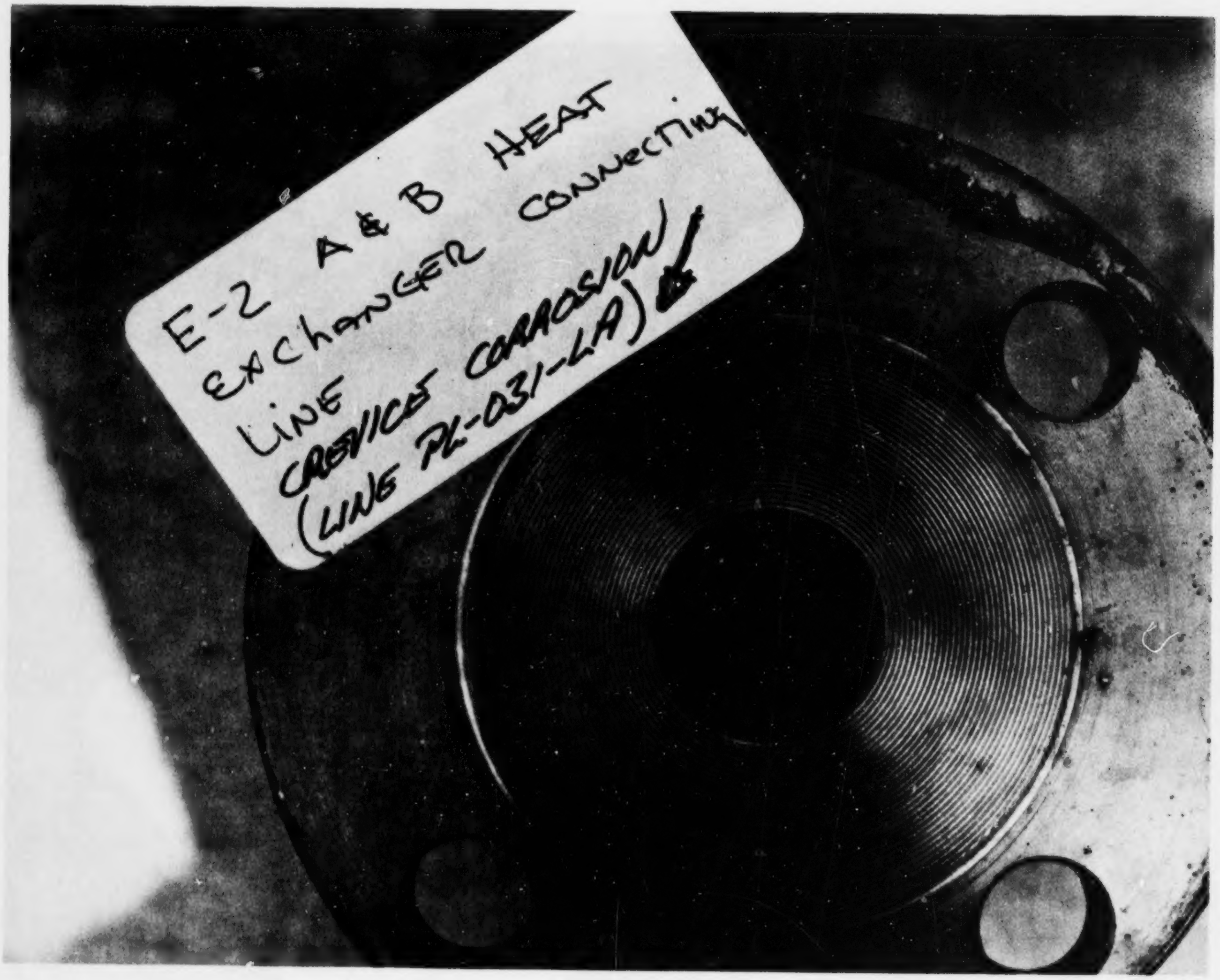

E-2 A & B HEAT
EXCHANGER CONNECTING
LINE
CREVICE CORROSION
(LINE PL-031-4A)

Figure 66. Flange Face on Process Line Mating with Heat Exchanger E-2 Outlet.

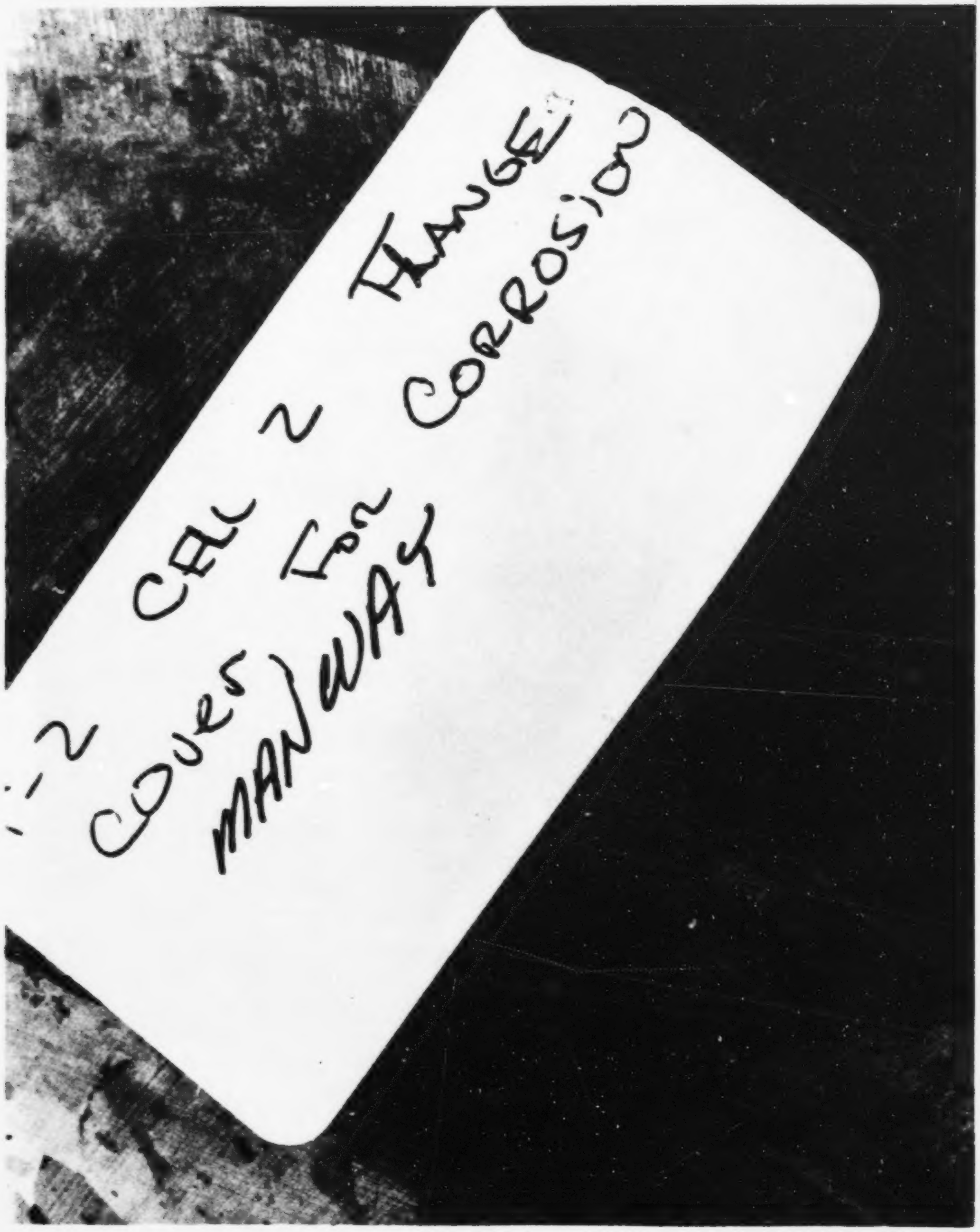

T-2 CELL 2 FLANGE
COVERS FOR CORROSION
MANWAY

Figure 67. Flange Face on Manway Port on Cell 2 of Mix Tank T-2.

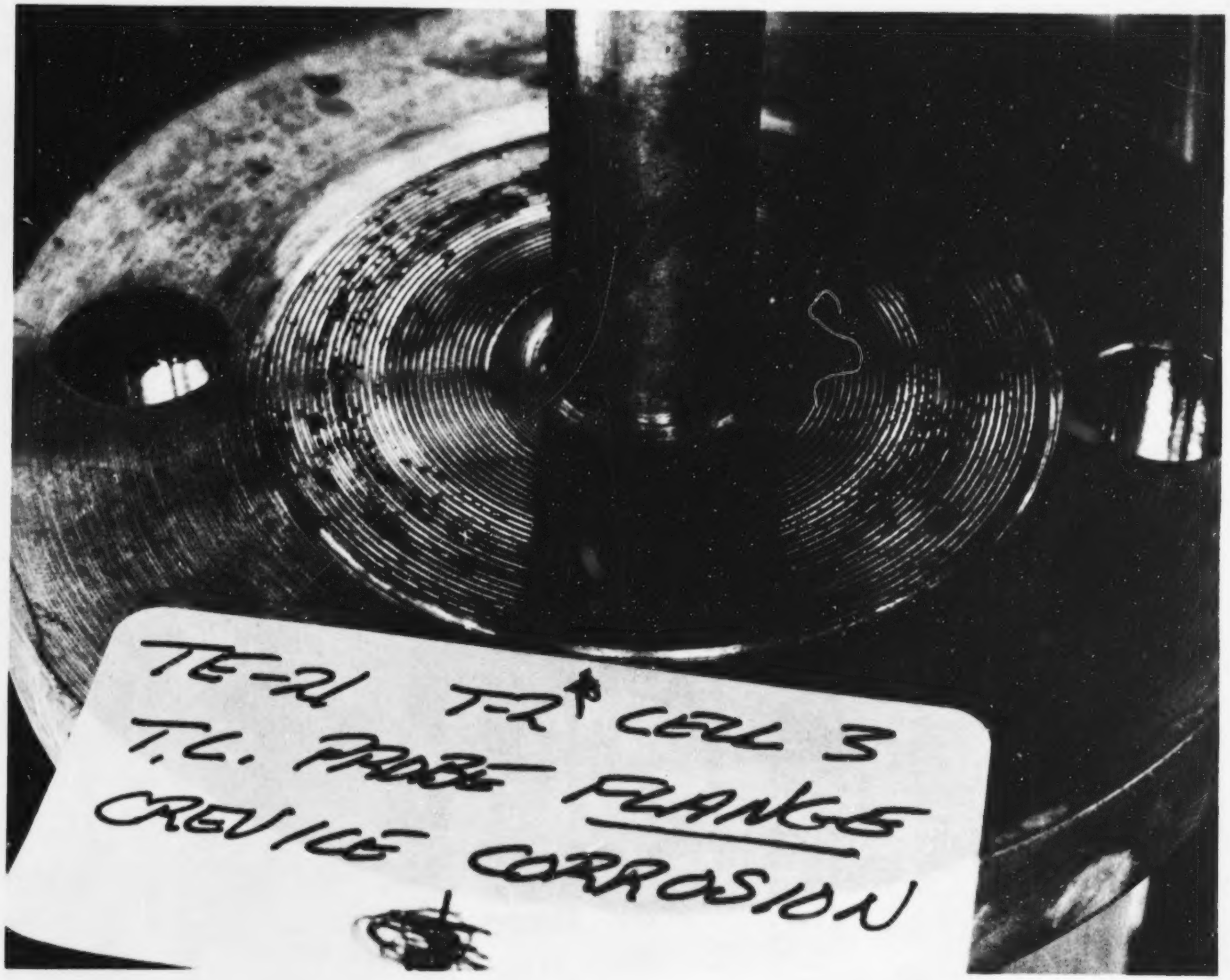

TE-21 T-2 CELL 3
T.C. PROBE FLANGE
CREVICE CORROSION

Figure 68. Thermocouple Probe TE-21 from Cell 3 of Mix Tank T-2.

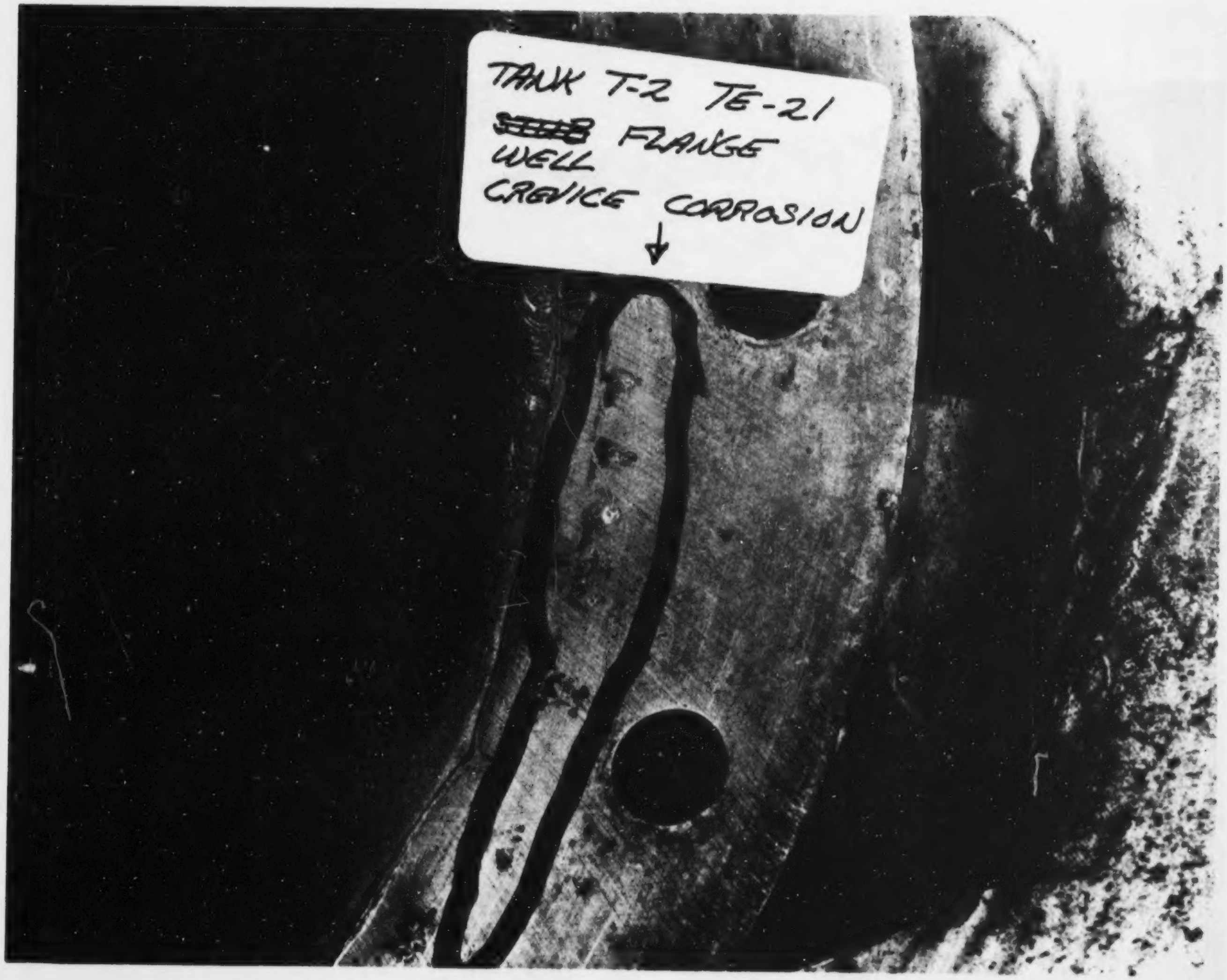

TANK T-2 TE-21
FLANGE
WELL
CREVICE CORROSION

Figure 69. Flange Face on Thermocouple Probe Port on Cell 3 of Mix Tank T-2.

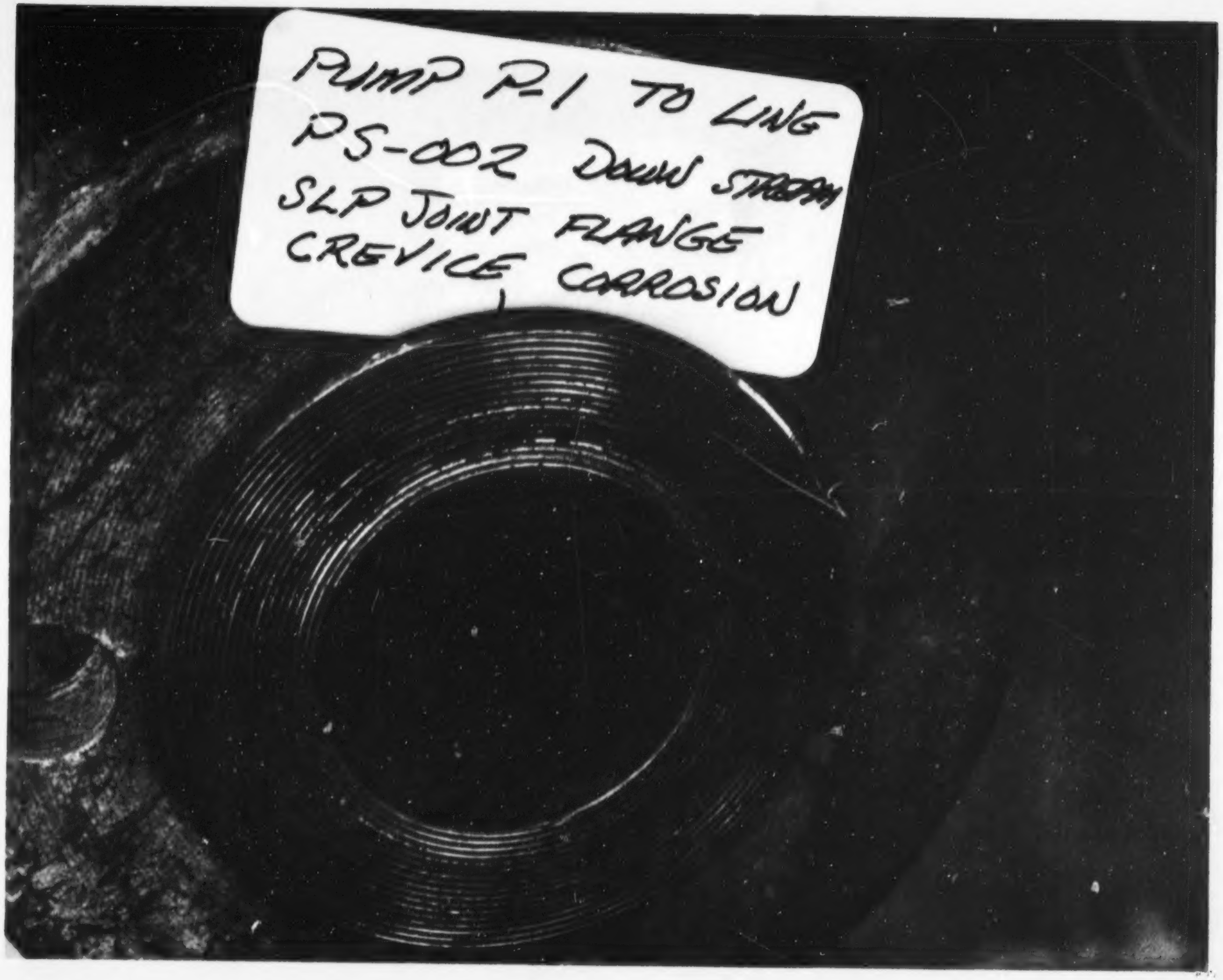

PUMP P-1 TO LINE
PS-002 DOWN STREAM
SLP JOINT FLANGE
CREVICE CORROSION

Figure 70. Flange Face on Outlet of Slurry Feed Pump P-1.

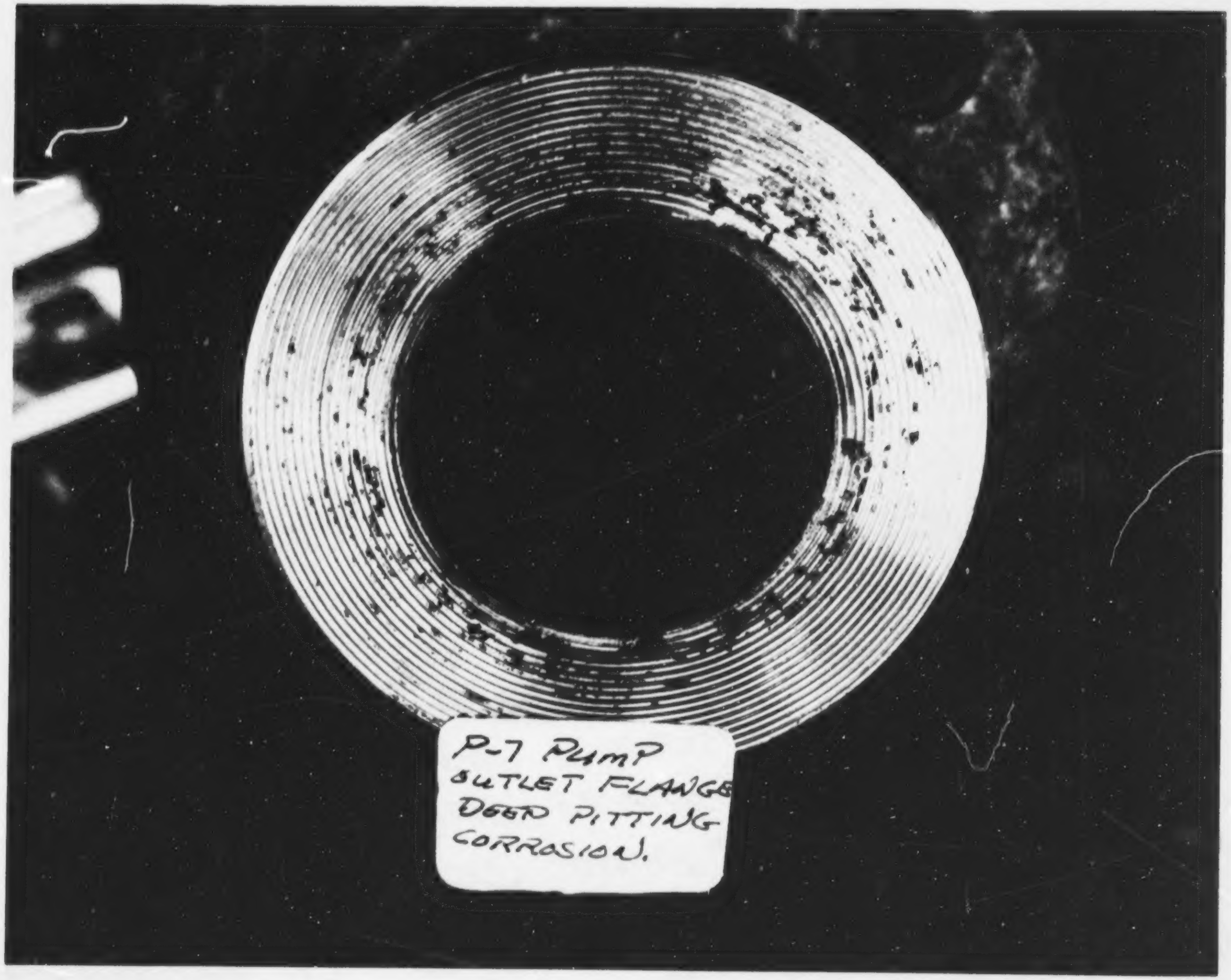

P-7 PUMP
OUTLET FLANGE
DEEP PITTING
CORROSION.

Figure 71. Flange Face on Outlet of Slurry Feed Pump P-7.

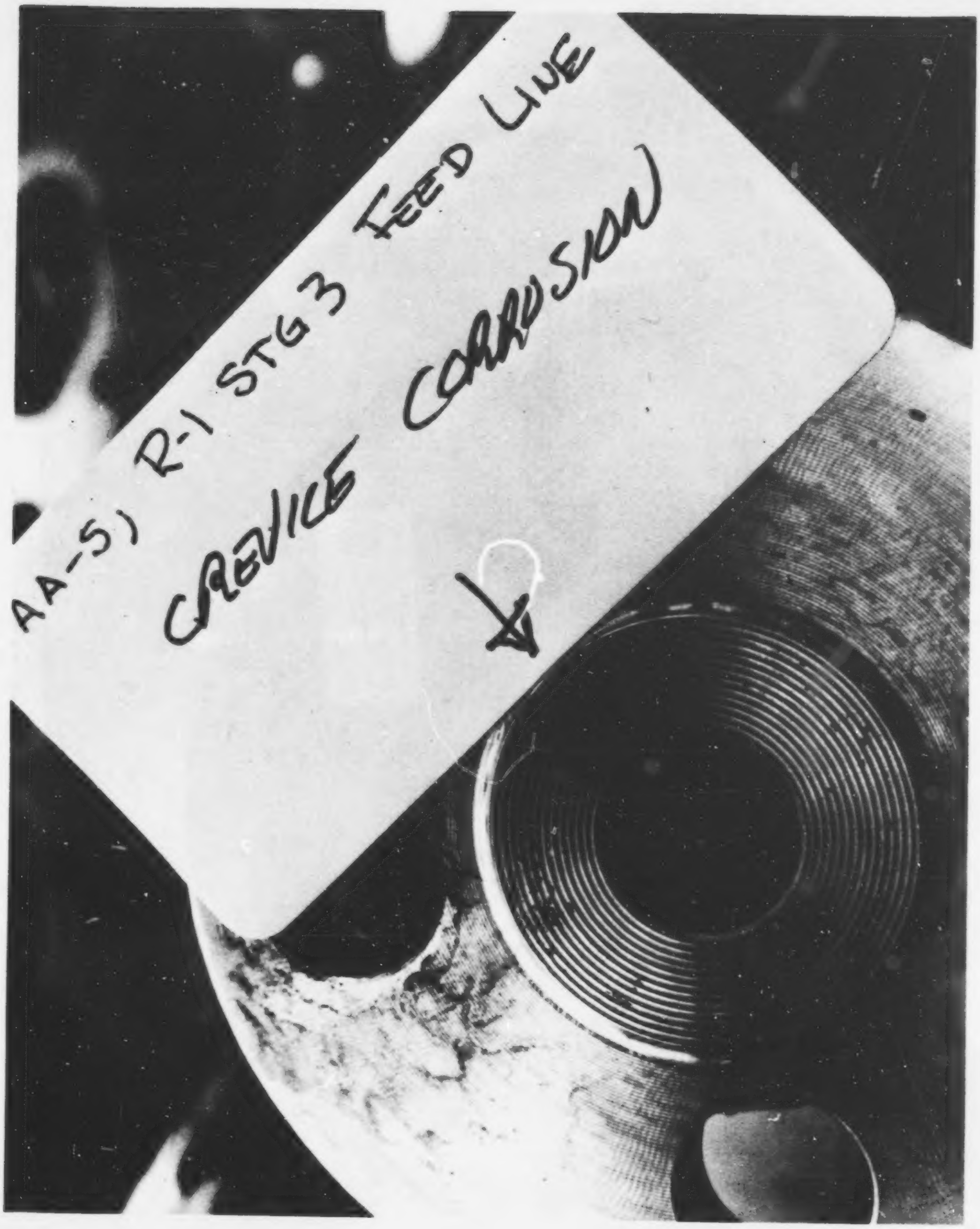

AA-5) R-1 STG 3 FEED LINE
CREVICE CORROSION

Figure 72. Flange Face from Slurry Feed Line to Cell 3 of Primary Reactor R-1.

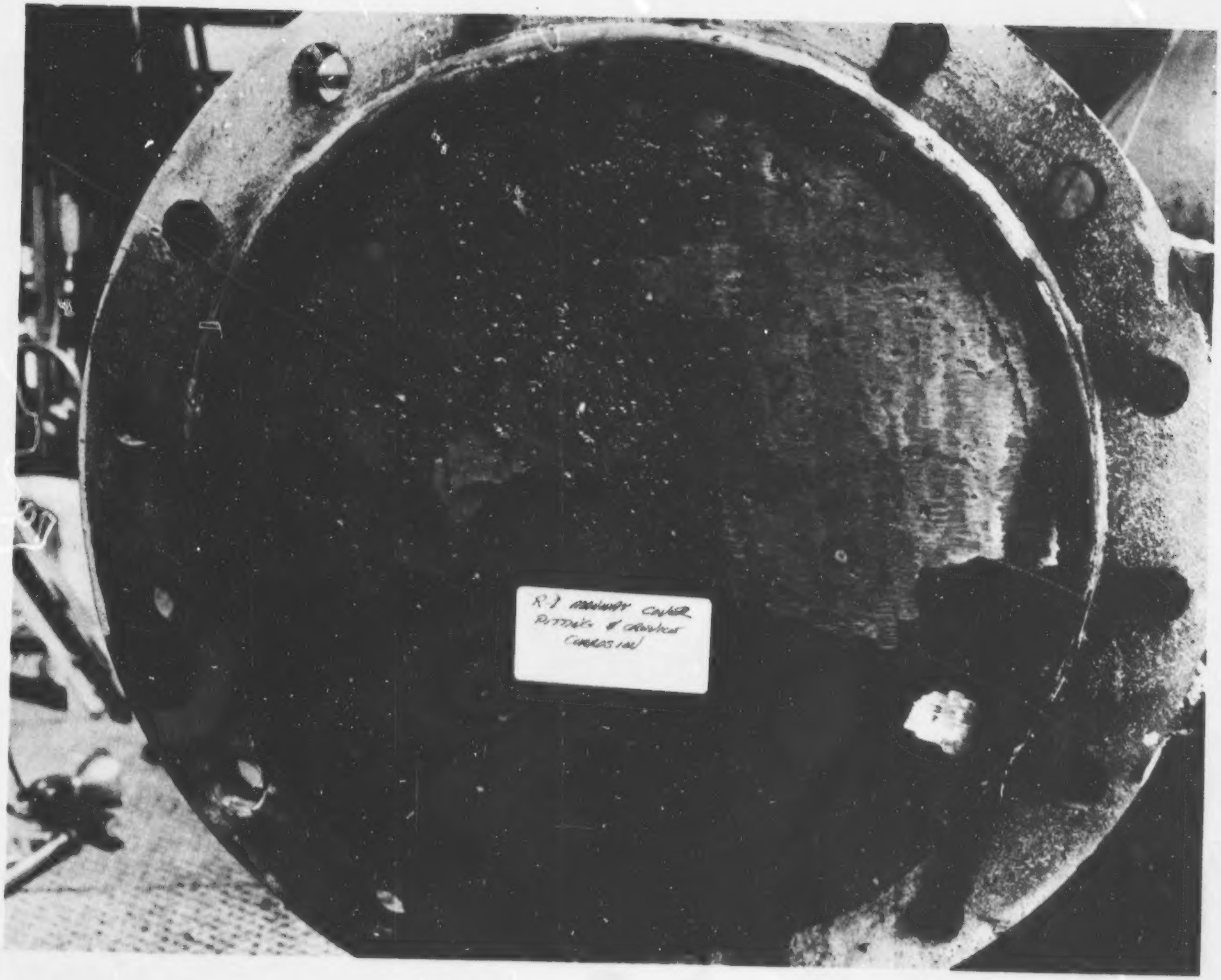

R-1 MANWAY COVER
PITTING & CREVICE
CORROSION

Figure 73. Manway Cover to Cell 3 of Primary Reactor R-1.

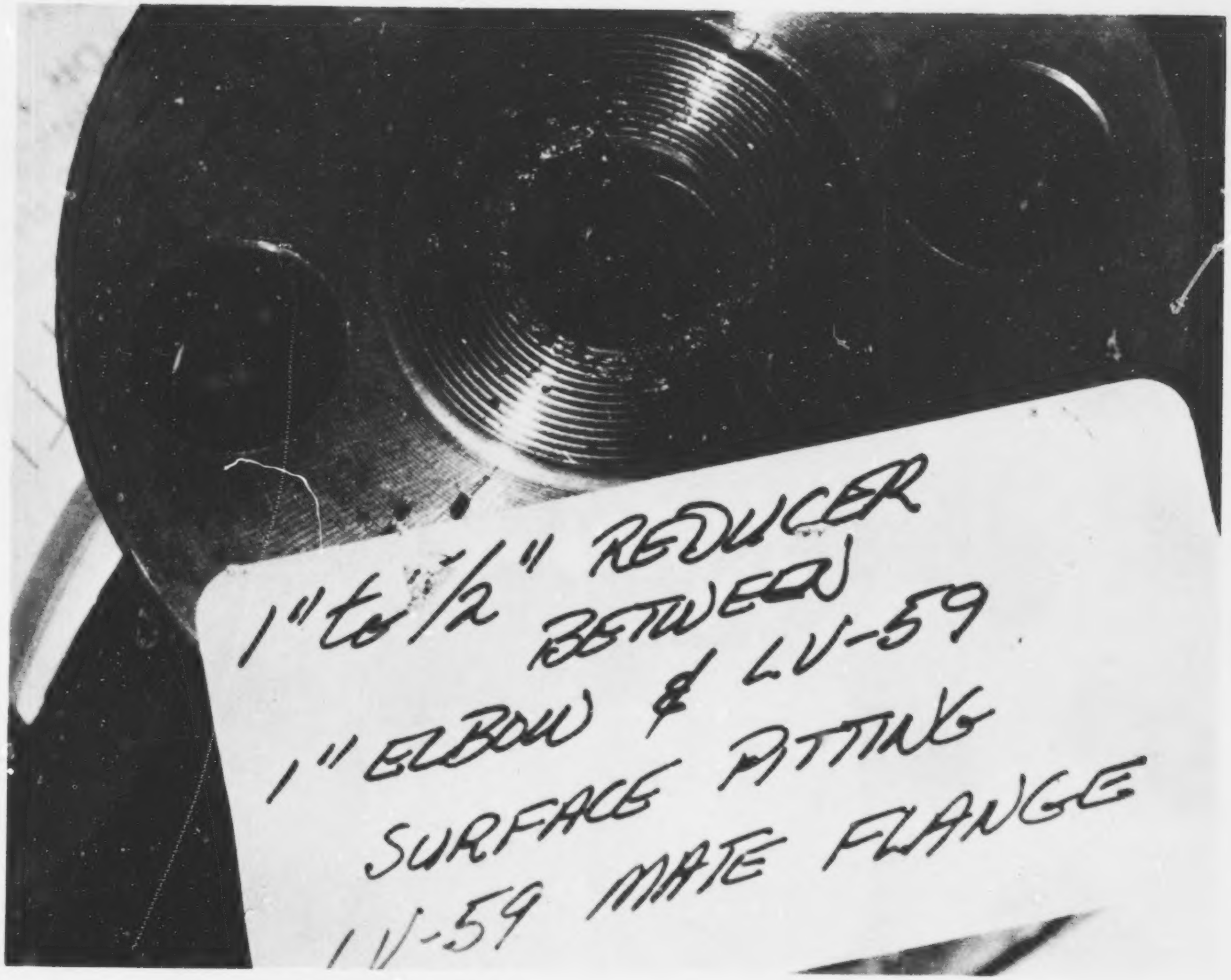

1" to 1/2" REDUCER
BETWEEN
1" ELBOW & LV-59
SURFACE PITTING
LV-59 MATE FLANGE

Figure 74. Face of Flange Mating with Low Level Control Valve LV-59.

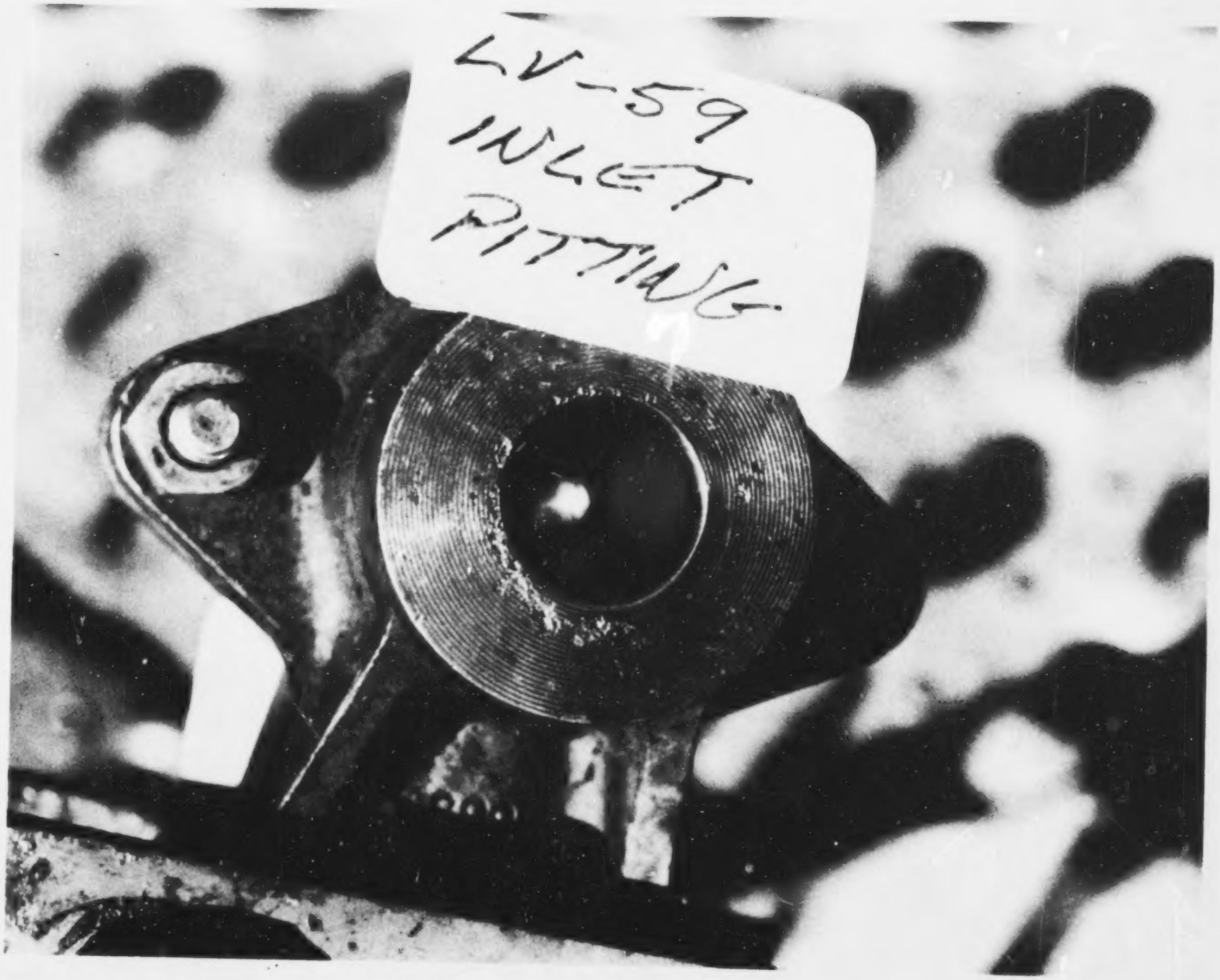

LV-59
INLET
PITTING

Figure 75. Flange Face on Inlet of Low Level Control Valve LV-59.

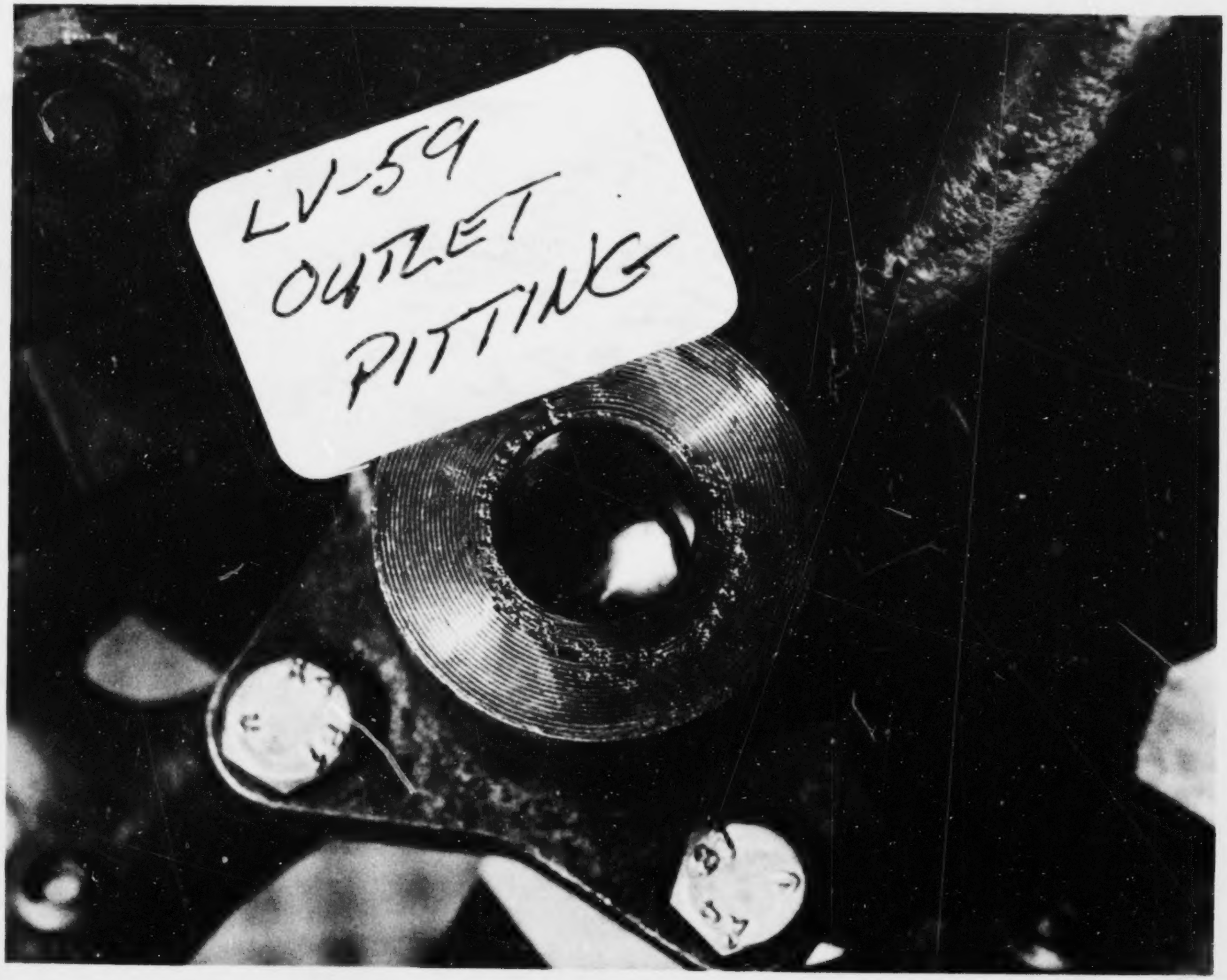
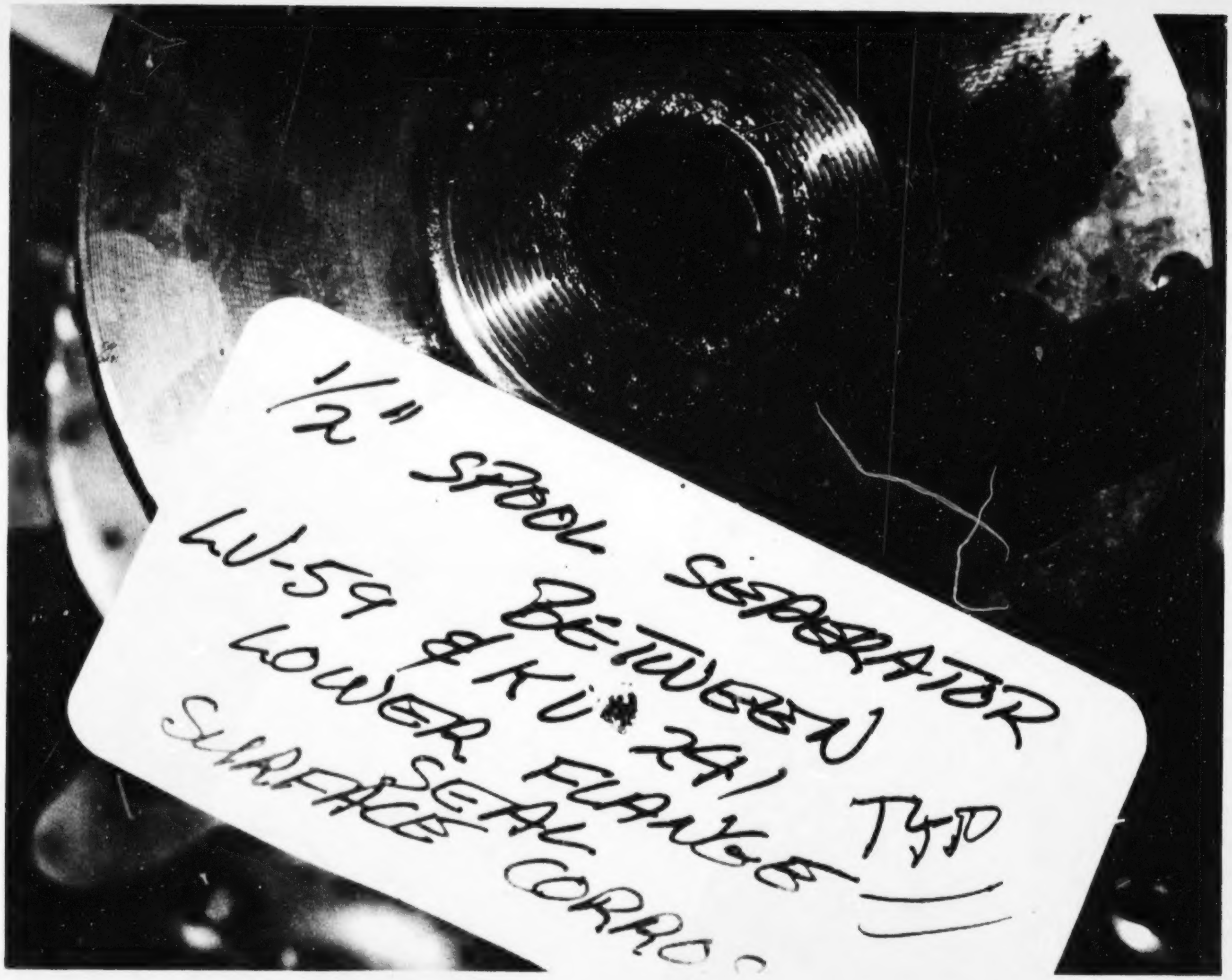

LV-59
OUTLET
PITTING

Figure 76. Flange Face on Outlet of Low Level Control Valve LV-59.



1/2" SPOOL SEPERATOR
BETWEEN
LV-59 & KV 241 TYP
LOWER FLANGE
SEAL
SURFACE CORRO

Figure 77. Flange Face on Spool Separating Low Level Control Valve LV-59 and Level Control Valve KV-241.

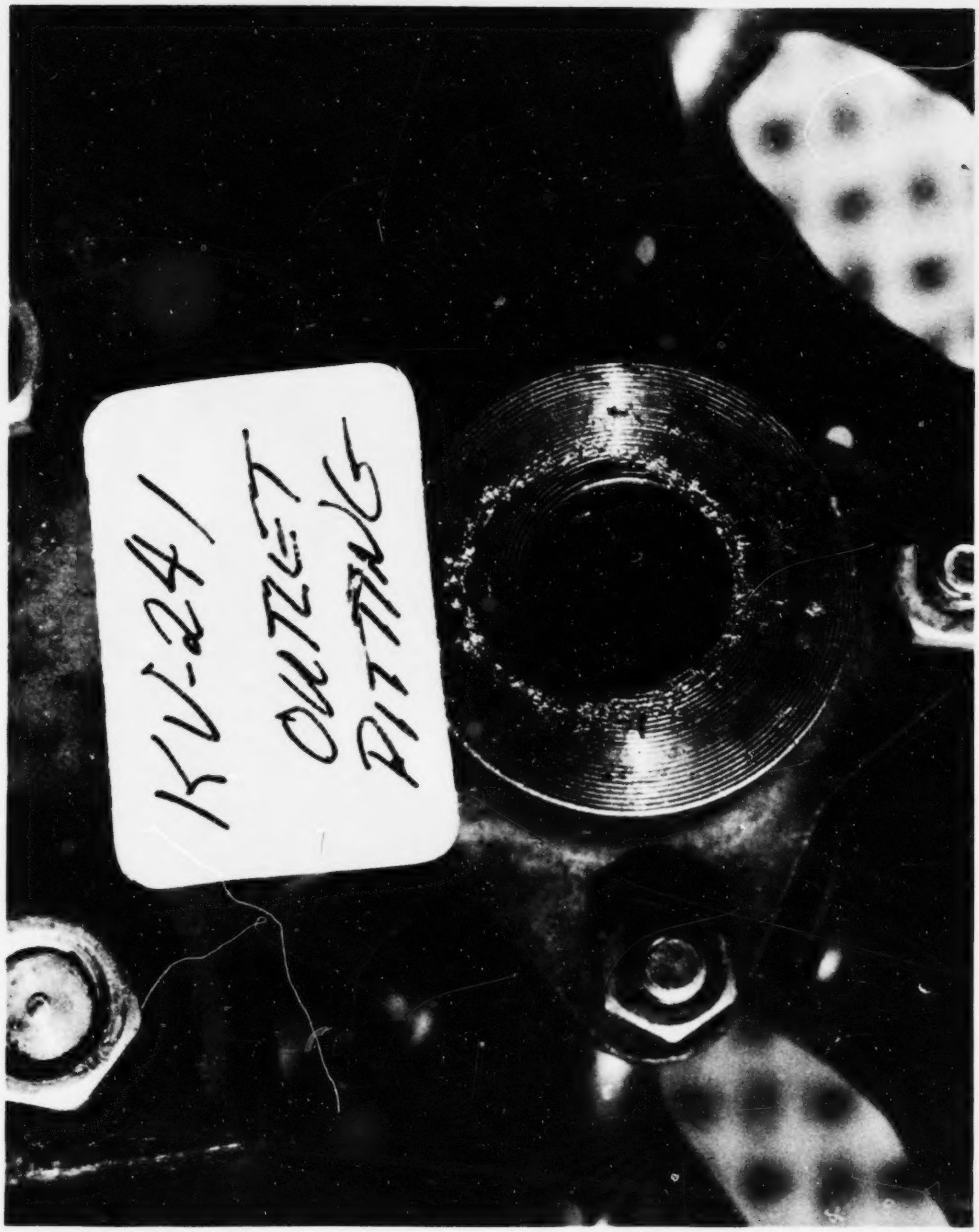

KV-241
OUTLET
PITTING

Figure 78. Flange Face on Inlet of Level Control Valve KV-241.

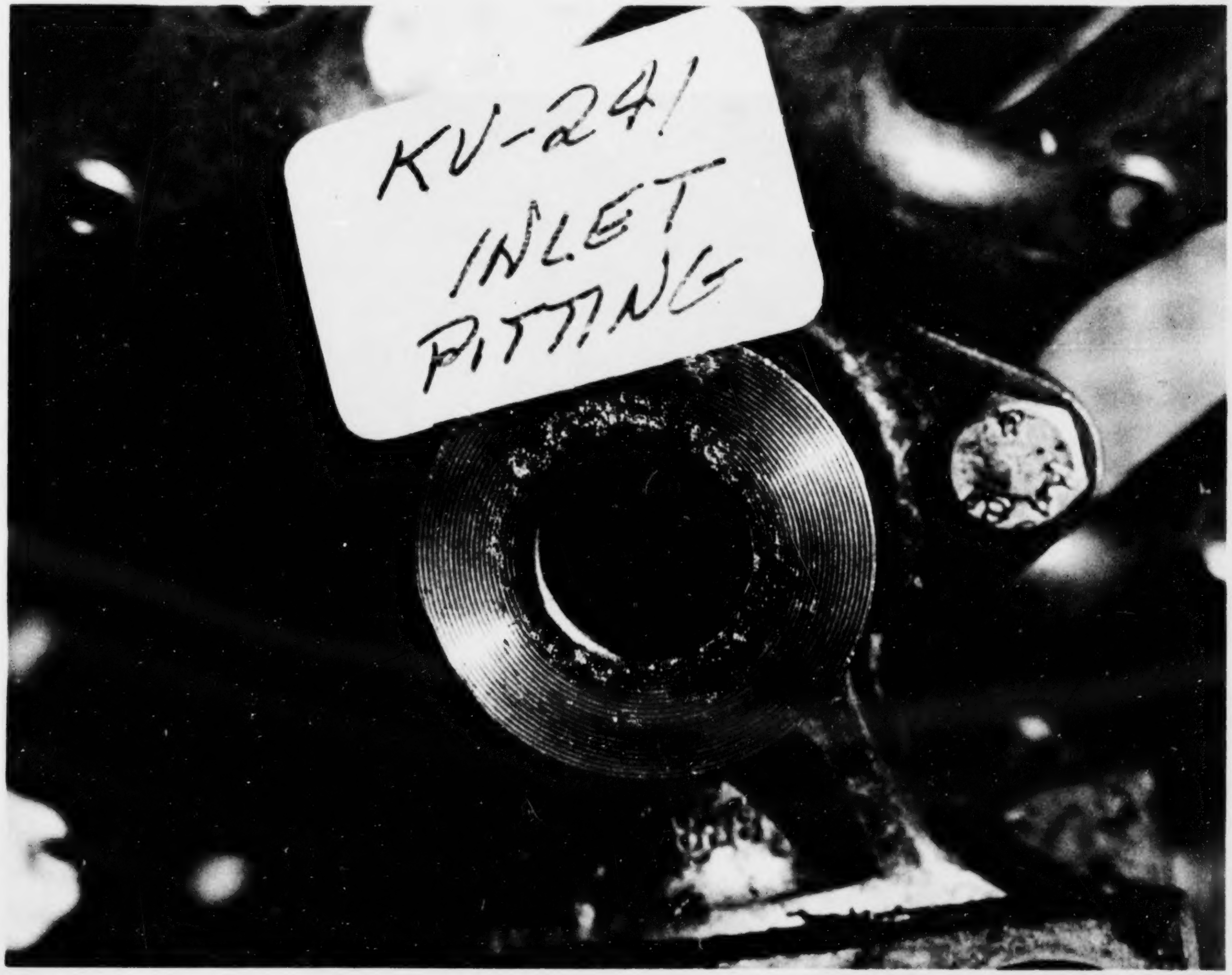

KV-241
INLET
PITTING

Figure 79. Flange Face on Outlet of Level Control Valve KV-241.

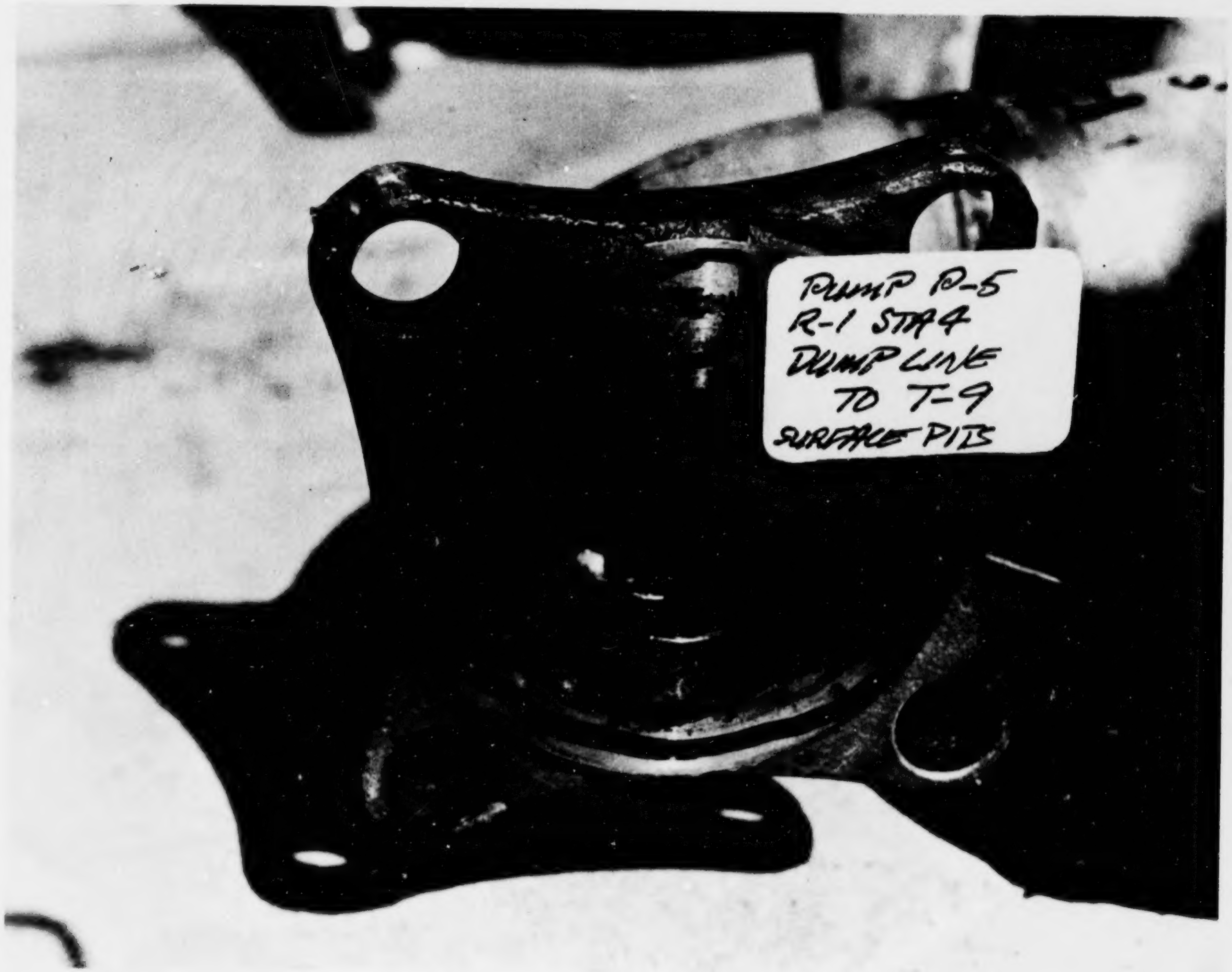

PUMP P-5
R-1 STA 4
DUMP LINE
TO T-9
SURFACE PITS

Figure 80. Flange Face on Reactor R-1 Cell 4 Discharge Line to Disposal Tank T-9.

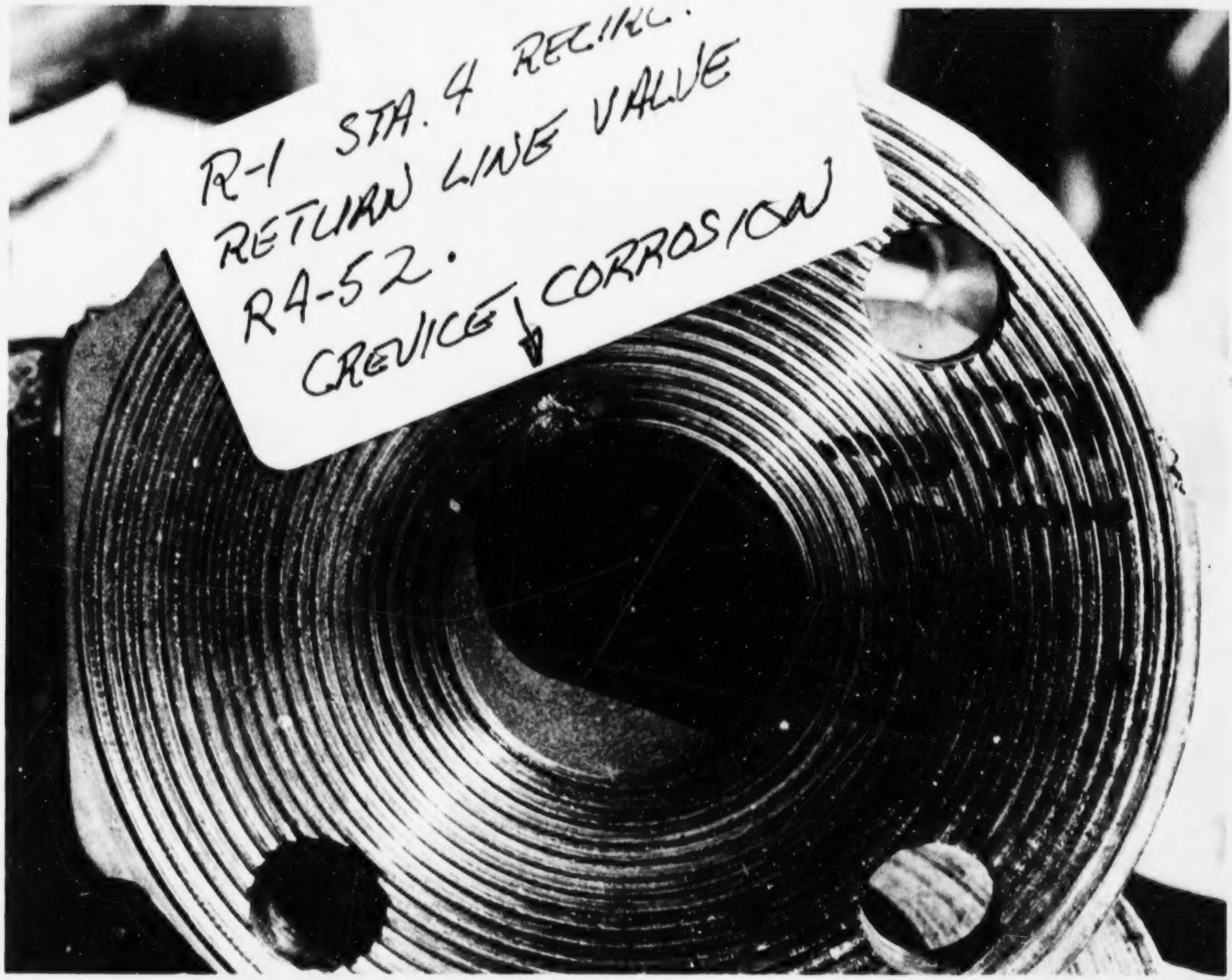

R-1 STA. 4 RECIRC.
RETURN LINE VALVE
R4-52.
CREVICE CORROSION

Figure 81. Flange Face on Valve R4-52 in Cell 4 Recirculation Loop for Reactor R-1.

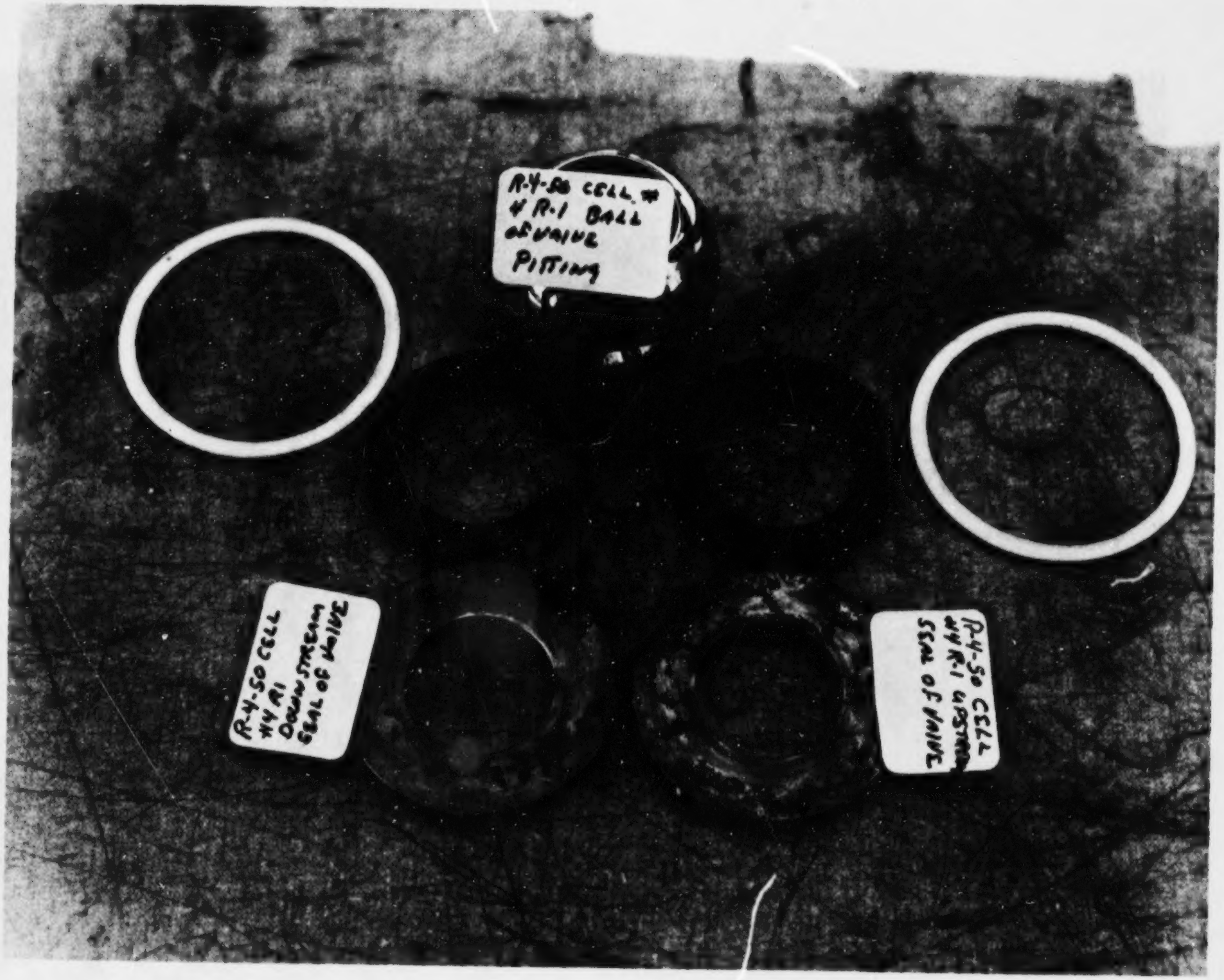

R-4-50 CELL # 4 R-1 BALL OF VALVE PITTING
R-4-50 CELL #4 R1 DOWN STREAM SEAL OF VALVE
R-4-50 CELL #4 R-1 UPSTREAM SEAL OF VALVE

Figure 82. Seal Retainer Rings and Ball for Valve R4-50 from Cell 4 Recirculation Loop for Reactor R-1.

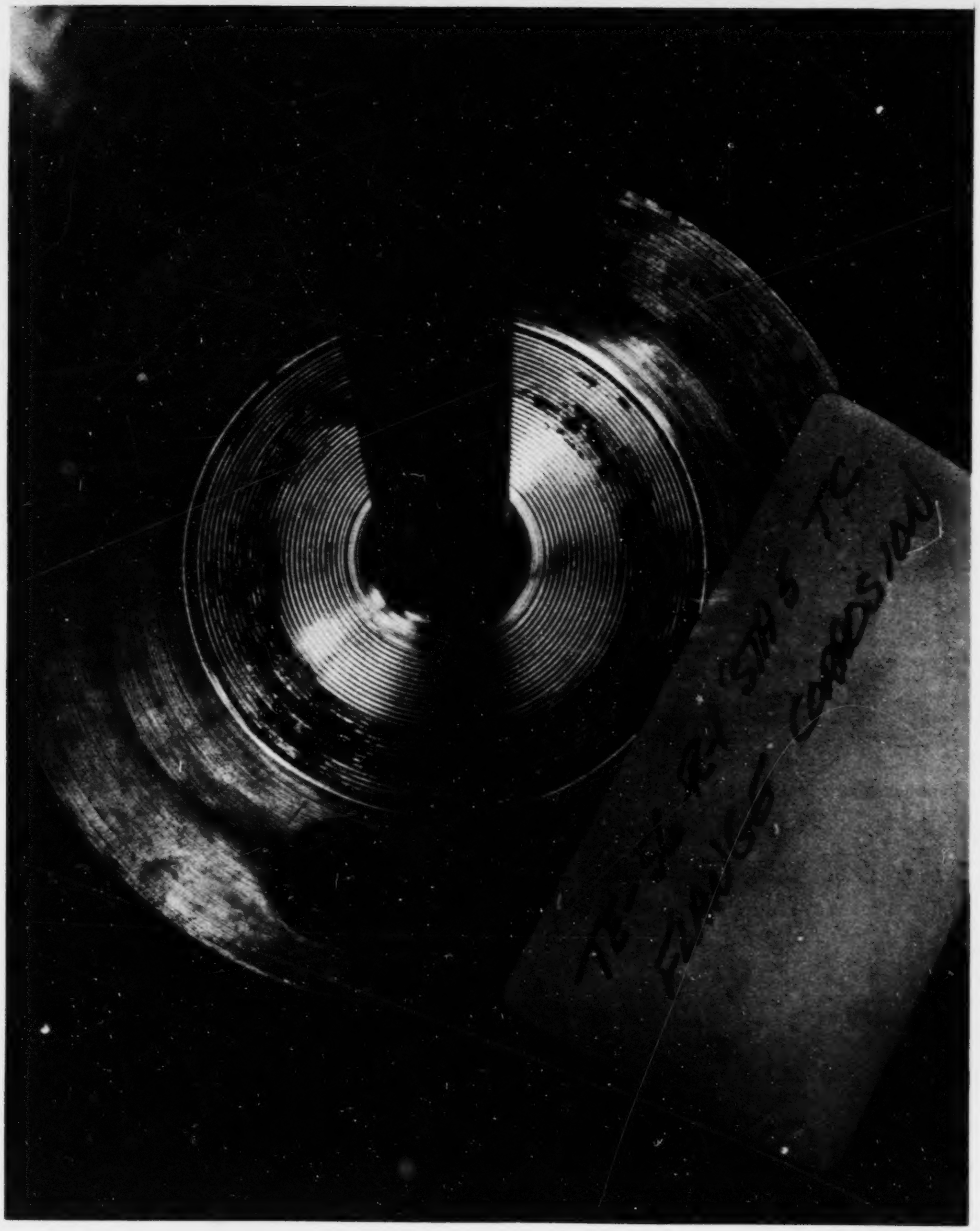

TE-56 R1 5TH'S TC
FLANGE CORROSION

Figure 83. Thermocouple Probe TE-56 from Cell 5 of Reactor R-1.

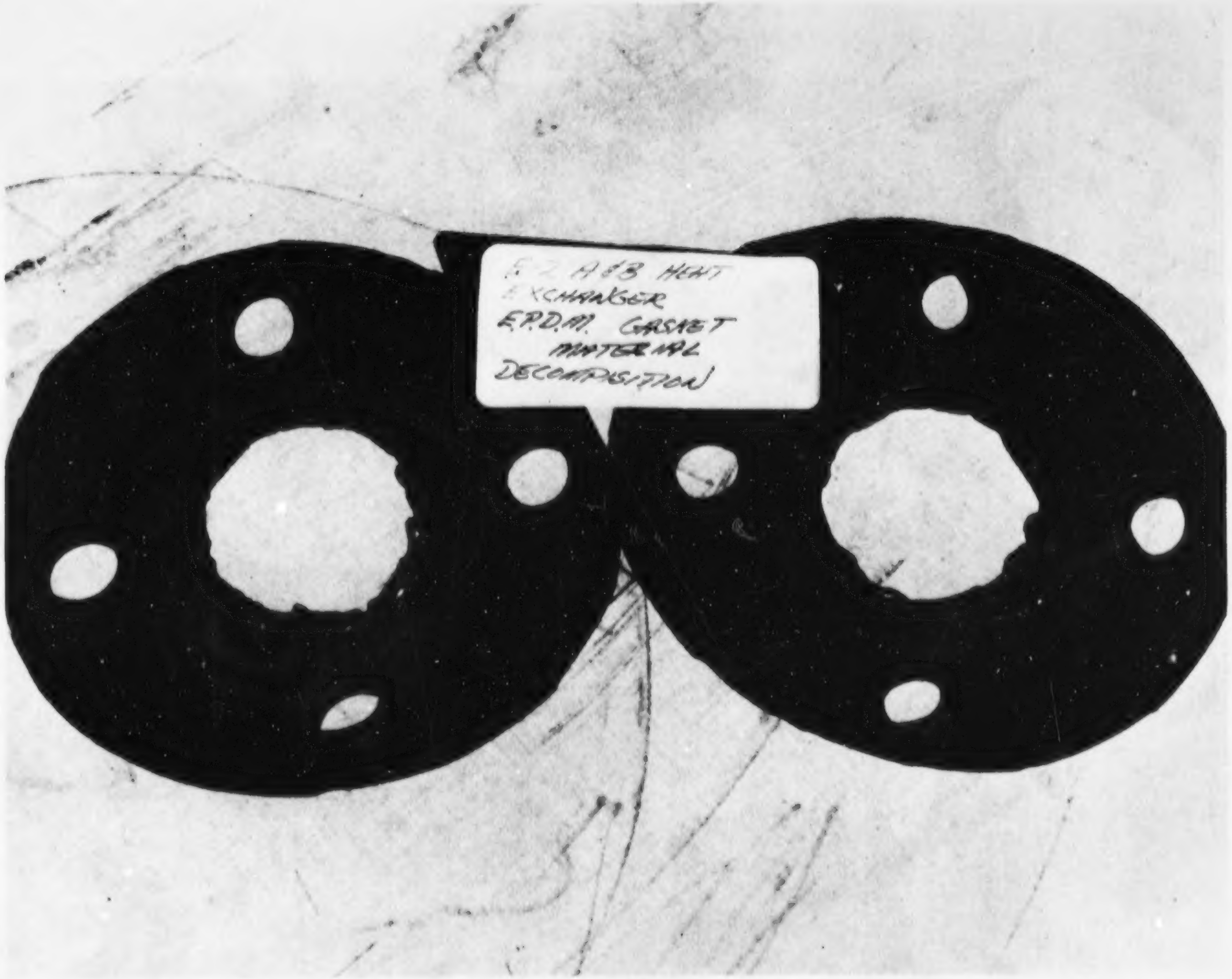

E-2 A&B HEAT
EXCHANGER
E.P.D.M. GASKET
MATERIAL
DECOMPOSITION

Figure 84. Flange Gaskets from Heat Exchanger E-2.

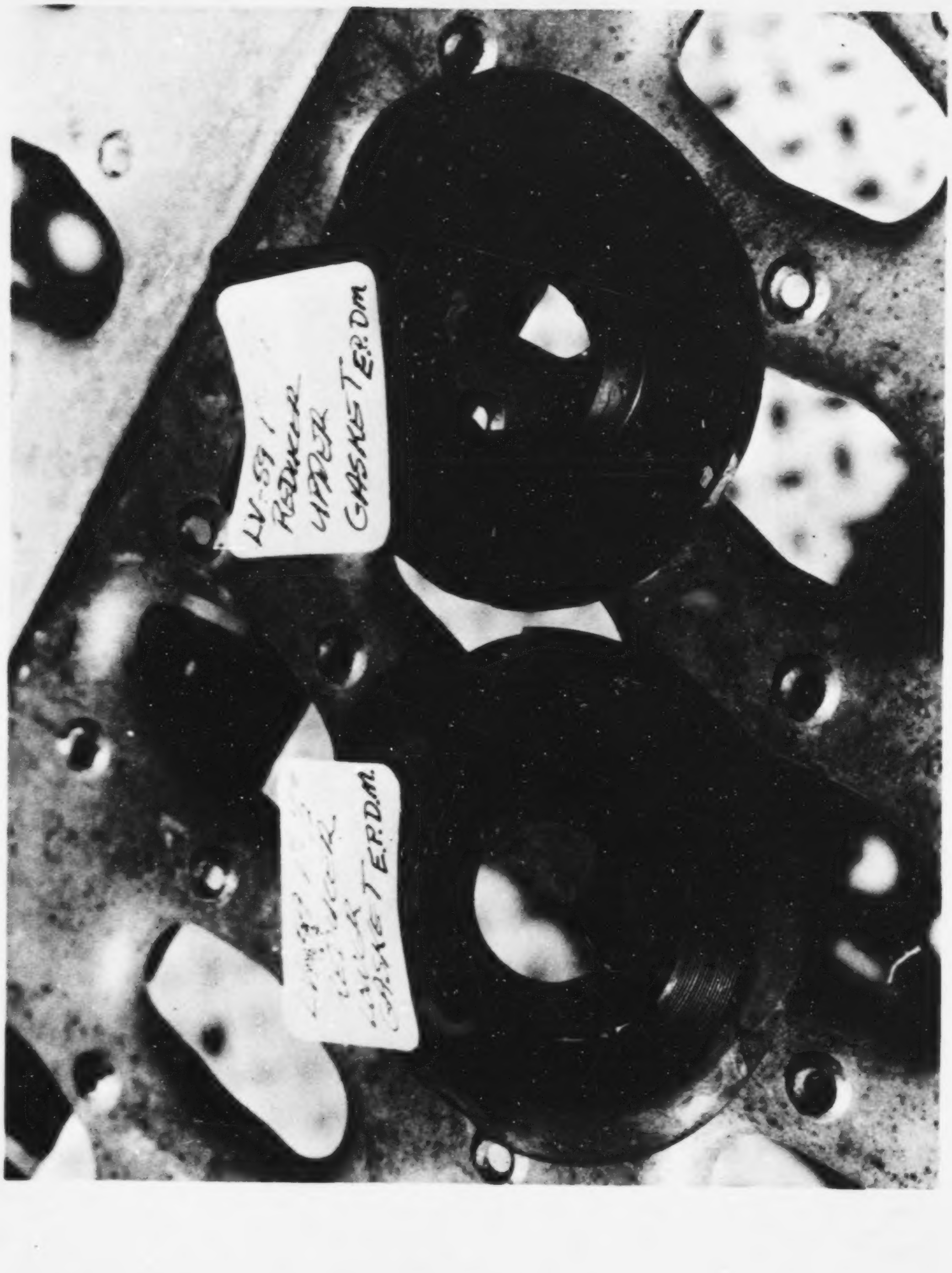

LV-59 REDUCER UPPER GASKET E.P.D.M
GASKET E.P.D.M.

Figure 85. Flange Gaskets from Low Level Control Valve LV-59.

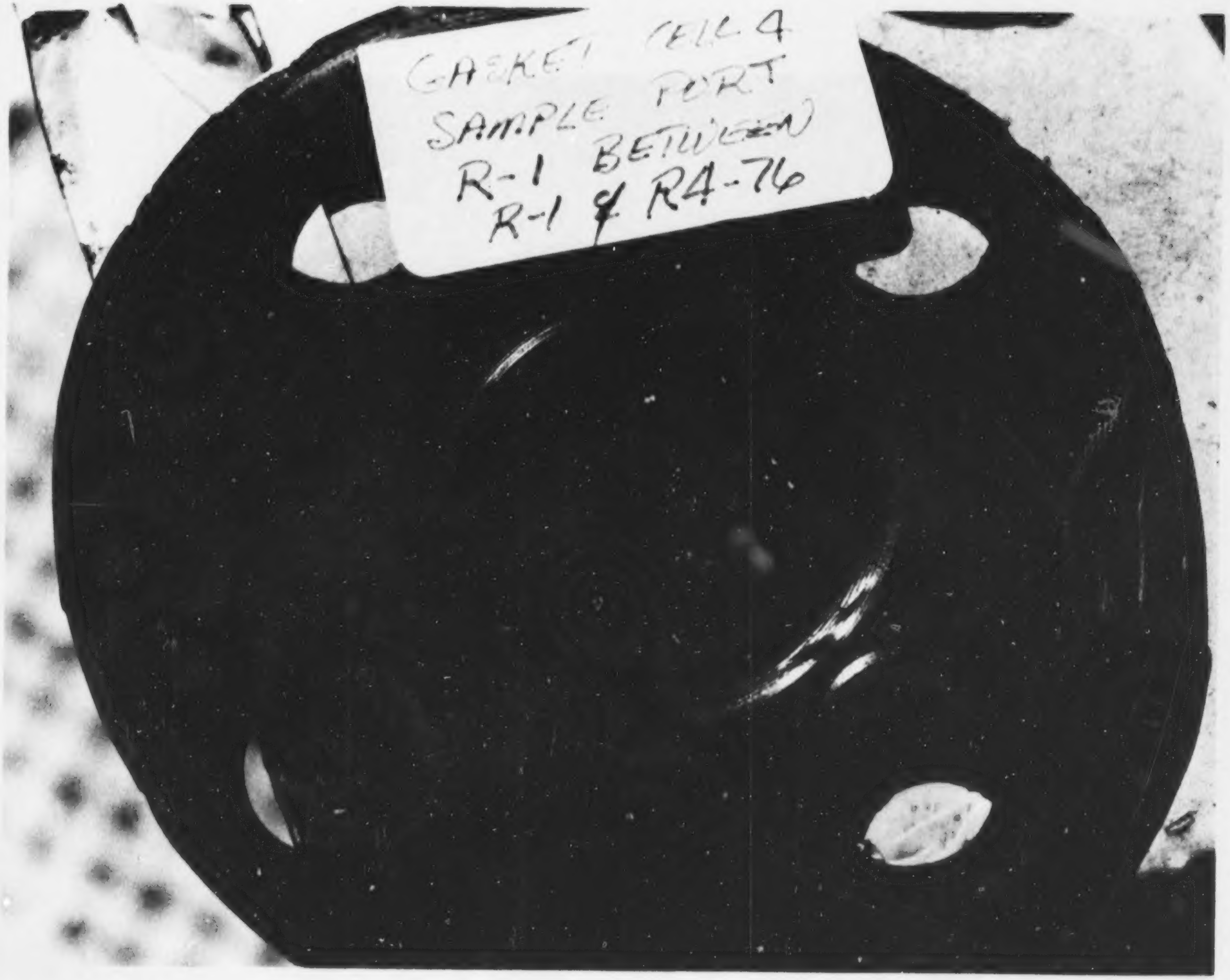

GASKET CELL 4
SAMPLE PORT
R-1 BETWEEN
R-1 & R4-76

Figure 86. Flange Gasket from Reactor R-1 Cell 4 Sample Valve R4-76.

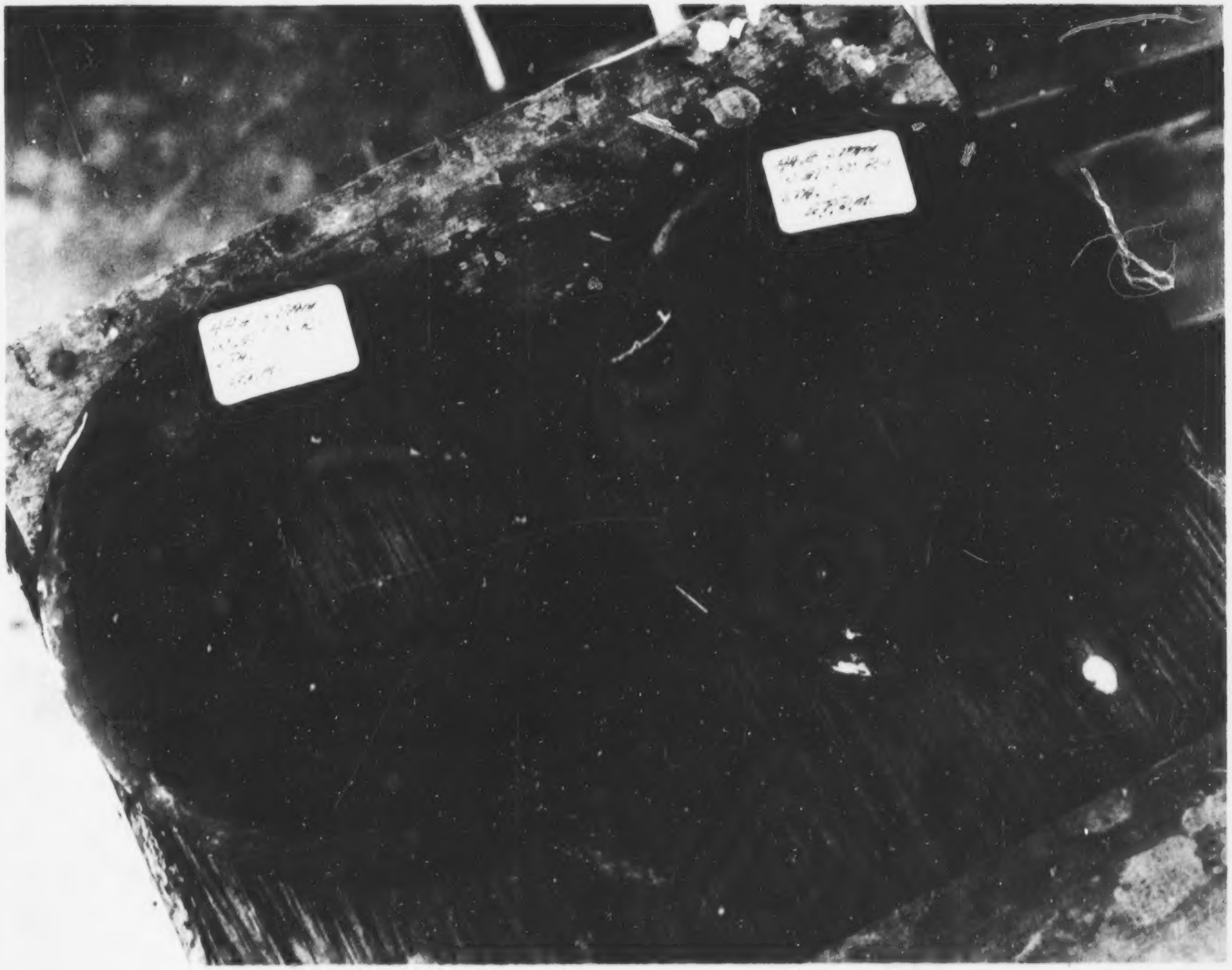


Figure 87. Flange Gaskets from Steam Inlet Line to Cell 1 of Reactor R-1.

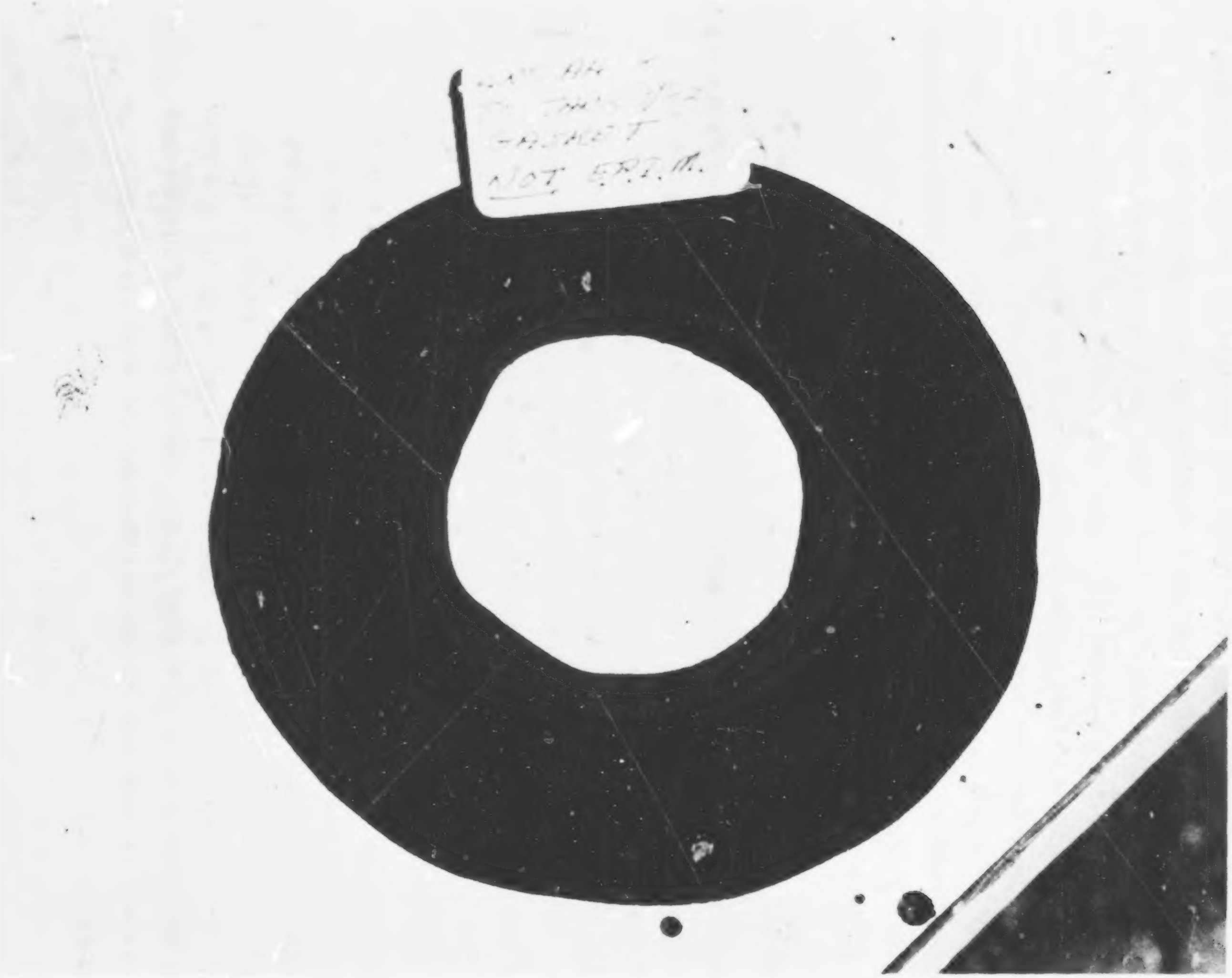

GASKET
NOT EPT.M.

Figure 88. Flange Gasket from Filtrate Receiver V-2 Discharge Line.

5.0 BENCH-SCALE EXTRACTIONS (TASK 2A)

A variety of coals were investigated for application of the Gravichem Process. The coals included two coals supplied by Tennessee Valley Authority (TVA) and five gob-pile coals. Each coal was separated by float-sink techniques in iron sulfate leach solution and/or conventional organic solvent at various top sizes. The resulting fractions were analyzed for sulfur forms, ash content and heat content after washing with water and drying. In selected cases the sink fraction, which contains most of the pyrite, was reached in leach solution according to Meyers Process conditions to chemically remove the pyrite. The sink fractions were analyzed for sulfur forms and heat and ash content.

5.1 TVA COAL STUDIES (TASK 2A1)

Kentucky No. 9 seam coal was supplied by the Tennessee Valley Authority for bench-scale extractions. Experimental investigations on the ROM and 3/8 x 0 coal were completed with respect to the float-sink separation of the coals in 1.3 specific gravity iron sulfate leach solution or organic liquid. Both float and sink fractions were analyzed for sulfur forms, ash content and heat content after washing with water and drying.

5.1.1 Float Sink (Organic Liquid) (Task 2A1a)

As-received run-of-mine (ROM) TVA coal of greater than 2 inch top size gave a 67% yield of float coal and a 33% yield of sink when gravity separated in 1.3 specific gravity organic liquid (Table 5). Gravity separation was accomplished at 20°C in a 1:4 coal and organic liquid slurry. The slurry was mixed thoroughly by careful agitation and then allowed to separate in a quiescent state. The resultant float product was carefully skimmed off of the solution and the sink subsequently recovered by filtration.

TABLE 5. SETTLING TANK FLOAT/SINK SEPARATION/ANALYSES OF TVA (AS RECEIVED) COAL

Separation Medium @ 20°C	Fraction	Yield %	Ash %, w/w	Heat Content, Btu	Sulfur Content, % w/w				Lbs SO_2
					Total	Pyrite	Sulfate	Organic	MM/Btu
--	Whole Coal	-	17.84	11577	3.84	2.80	0.44	0.61	6.63
Organic* Liquid (1.3 S.G.)	Float	67	7.17	13423	2.56	1.00	0.06	1.50	3.81
	Sink	33	36.82	8590	6.05	4.33	0.77	0.95	14.09
Leach** Solution (1.3 S.G.)	Float	43	4.70	13833	2.36	0.71	0.01	1.64	3.41
	Sink	57	25.54	10377	4.19	3.41	0.50	0.28	8.08
(Reacted Sink)***		-	18.98	11486	3.35	1.48	0.41	1.46	5.83

[*] Mixture of toluene and perchloroethylene, wt ratio 1:1.35.

[**] Aqueous ferric sulfate (ferri-floc) with specific gravity obtained as follows:

Fe^{+++} as $Fe_2(SO_4)_3$ (wt %)	H_2SO_4 (wt %)	H_2O and $SO_4^{=}$ as $Fe_2(SO_4)_3$ (wt %)
~ 7.5	~ 4.0	~ 88.5

[***] Reacted for 48 hours @ 102°C in aqueous ferric sulfate (ferri-floc) above**.

5.1.2 Float-Sink (Leach Solution) (Task 2A1b)

ROM Kentucky No. 9 coal greater than 2 inch top-size gravity separated at 20°C in 1.3 specific gravity Meyers Process leach solution (7.5% Fe, 4% H_2SO_4). A 1:4 coal and leach solution slurry was mixed thoroughly by careful agitation and then allowed to gravity separate in a quiescent state. The resultant float product was carefully skimmed off of the solution and the sink subsequently recovered by filtration. The product coals (float and sink) were washed and dried and the yields determined, i.e., 43% float and 57% sink (Table 5). This compares favorably with the yield obtained using 3/8" x 0 top-size Tennessee Valley Authority coal (40% float, 60% sink) reported in Section 2A1d.

5.1.3 Meyers Process on ROM Coal (Sink) (Task 2A1c)

The sink fraction of the ROM Kentucky No. 9 coal from the 1.3 specific gravity Meyers Process leach solution (Scope-Section 2A1b) was treated by the Meyers Process for 48 hours at 102°C. It is seen in Table 5 that a significant reduction in ash and pyritic sulfur was attained, however, the product sink coal does not meet the "4-lb" state standard after processing for the stated 48-hour period.

5.1.4 Float-Sink 3/8" x 0 Coal (Task 2A1d)

ROM Kentucky No. 9 seam coal supplied by the Tennessee Valley Authority was subjected to float-sink separation in 1.3 specific gravity iron sulfate leach solution or organic liquid at 3/8" x 0 top-size. Both float and sink fractions were analyzed for particle size distribution, sulfur forms, ash content and heat content after washing with water and drying. Analyses are presented in Tables 6 and 7.

The particle size distribution data (Table 6) shows that there is no significant particle size segregation in either leach solution or organic solvent float-sink techniques. The analytical data in Table 7 shows that organic solvent and leach solution gravity separation give near identical products and that the Gravichem Process will give a 40/60 float vs sink product yield.

TABLE 6. PARTICLE SIZE OF FLOAT AND SINK FRACTIONS OF TVA COAL

Screen Size	% Retained Organic Float Fraction	Leach Float Fraction	Organic Sink Fraction
3/8 inch	2.6	1.5	2.2
1/4 inch	26.7	22.0	22.6
4 mesh	16.9	17.6	14.4
8 mesh	23.4	24.6	19.7
14 mesh	13.5	14.8	12.2
48 mesh	12.6	15.0	15.3
Pan	4.3	4.5	13.6
TOTAL	100.0	100.0	100.0

The Tennessee Valley Authority sink coal from the leach solution separation was size-reduced and treated by the Meyers Process to the 90% pyrite removal level (Table 8). The recovered sink fraction resulting from the Gravichem Process (Figure 89) was crushed by blending a 1:3 coal and leach solution (7.5% w/w Fe and 4% H_2SO_4; specific gravity 1.3) slurry in a one-gallon stainless steel laboratory blender for 10 minutes at ∿ 15,000 rpm. A temperature rise from 20°C to 70°C was noted in the resultant blend during the stated residence time. The remaining slurry was filtered and the coal cake retained for further processing.

Particle size analysis of a representative sample of the coal cake was performed and it is seen in Figure 90 that significant size reduction is attained for the sink fraction ground in the leach solution.

A series of extractions was performed on the ground sink fraction at 102°C (atmospheric pressure) with a 1.3 specific gravity solution containing 7.5% w/w Fe and 4% H_2SO_4. Processing was accomplished at intervals up to 48 hours. Data from this experimentation are presented in Table 8.

TABLE 7. FLOAT/SINK SEPARATION/ANALYSES OF TVA COAL

Separation Medium @ 20°C	Fraction	Yield %	Ash %, w/w	Heat Content, Btu	Sulfur Content, % w/w				$\frac{\text{lbs } SO_2}{\text{MM Btu}}$
					Total	Pyrite	Sulfate	Organic	
--	Whole Coal	--	19.14	11675	3.78	2.08	0.11	1.59	6.48
Organic* Liquid (1.3 S.G.)	Float	39	4.90	13851	2.37	0.56	0.05	1.76	3.42
	Sink	61	29.6	9868	4.76	3.16	0.23	1.37	9.65
Leach** Solution (1.3 S.G.)	Float	40	3.97	13923	2.30	0.59	0.01	1.70	3.30
	Sink	60	26.96	10731	4.94	3.19	0.35	1.40	9.21

* Mixture of toluene and perchloroethylene, wt ratio 1:1.35

** Aqueous ferric sulfate (Ferri-floc) with specific gravity obtained as follows:

Fe^{+++} as $Fe_2(SO_4)_3$ (wt %)	H_2SO_4 (wt %)	H_2O and $SO_4^{=}$ as $Fe_2(SO_4)_3$ (wt %)
~ 7.5	~ 4.0	~ 88.5

TABLE 8. RATE DATA ON PROCESSING 1.3 S.G. TVA COAL SINK FRACTION WITH 7.5% IRON SOLUTION ($4\%\ H_2SO_4$) AT 102°C AND ATMOSPHERIC PRESSURE

Exper. No.	Process Time Hrs.	Ash % w/w	Heat Content Btu/lb	Sulfur Content, % w/w				$\frac{\text{lbs } SO_2}{\text{MM Btu}}$
				Total	Pyrite	Sulfate	Organic	
Starting Coal (1.3 sink)								
Leach Solution	-	26.96	10731	4.94	3.19	0.35	1.40	9.21
Organic Liquid	-	29.58	9875	4.76	3.17	0.23	1.37	9.64
1	2	22.33	11188	3.48	1.85	0.37	1.26	6.22
2	4	20.80	11424	2.97	1.38	0.20	1.38	5.20
3	8	19.97	11540	2.69	0.93	0.28	1.47	4.66
4	14	18.89	12038	2.32	0.57	0.28	1.48	3.85
5	48	17.42	11544	2.32	0.33	0.36	1.63	4.02
Whole Coal (Prior to gravity separation)	-	19.14	11675	3.78	2.08	0.11	1.59	6.48

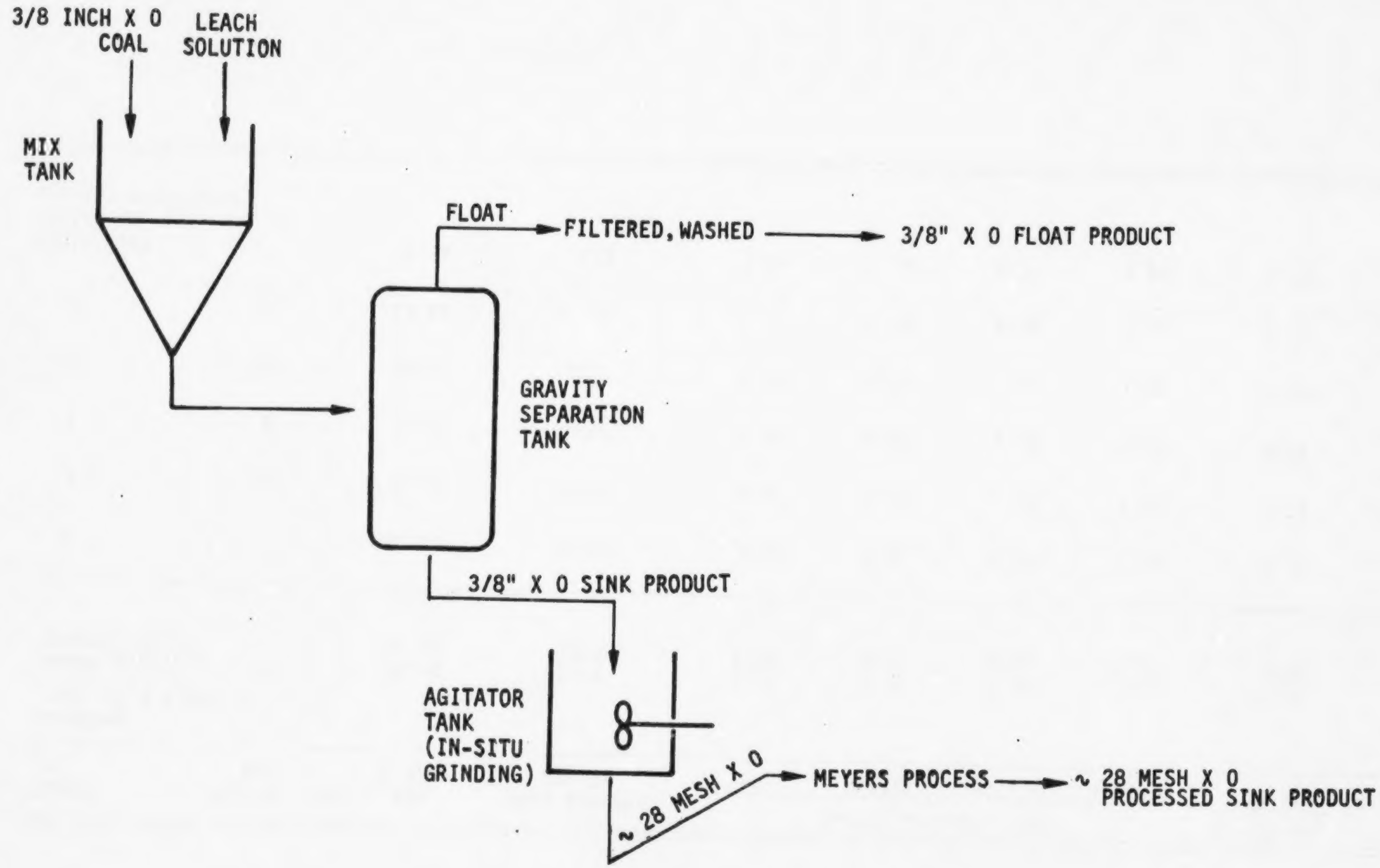

3/8 INCH X 0
COAL
LEACH
SOLUTION
MIX
TANK
FLOAT
FILTERED, WASHED
3/8" X 0 FLOAT PRODUCT
GRAVITY
SEPARATION
TANK
3/8" X 0 SINK PRODUCT
AGITATOR
TANK
(IN-SITU
GRINDING)
~ 28 MESH X 0
MEYERS PROCESS
~ 28 MESH X 0
PROCESSED SINK PRODUCT

Figure 89. Gravichem Processing of TVA Coal.

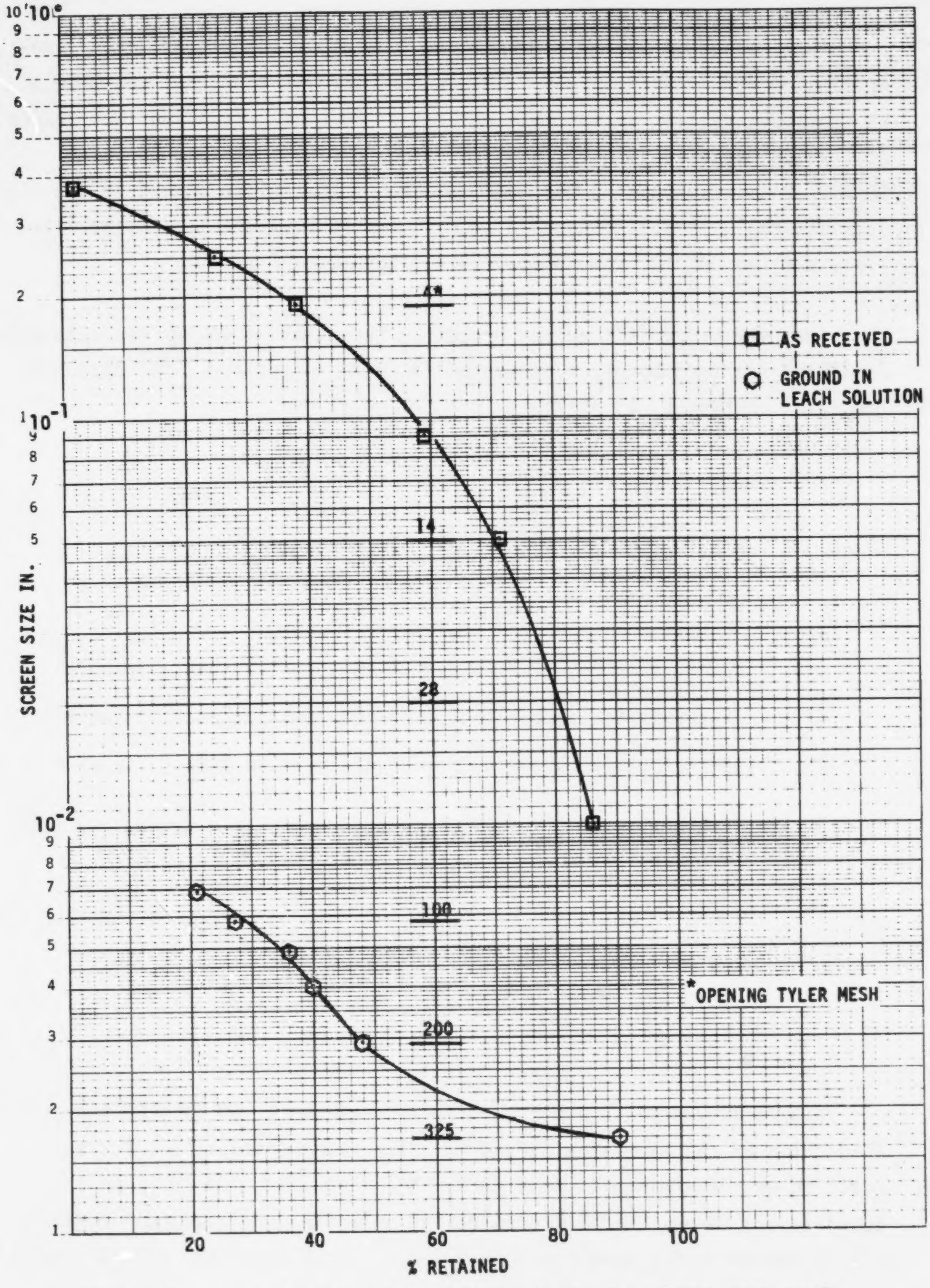


Figure 90. Size Distribution by Sieve Analysis of TVA Sink Coal.

Data presented in Table 8 indicate that ambient pressure Meyers processing of ROM Kentucky No. 9 1.3 specific gravity sink coal can effect at least 90% pyritic sulfur (S_p) removal in 48 hours resulting in a product coal which meets the Tennessee State Standard requirements of 4 lbs $SO_2/10^6$ Btu. Considering the high ash (27.0) and sulfur (4.9%) of the sink starting coal, this represents a significant improvement in coal quality; i.e., 9.2 lbs $SO_2/10^6$ Btu reduced to 4.0 lbs $SO_2/10^6$ Btu.

The rate of pyritic sulfur reduction in the above series was determined using the standard approach presented in earlier studies(1,2). Basically, the rate constant K_L is obtained from the relation:

$$K_L = \frac{\frac{1}{W_p} - \frac{1}{W_p^\circ}}{t_{Y}^{-2}} \qquad (1)$$

where

t - Is the time required to reduce coal pyrite to W_p, in hours

W_p° - Is the pyrite concentration of the starting coal, in wt %

W_p - Is the pyrite concentration of the coal at time t, in wt %

$\bar{Y}$ - Is the average ferric ion-to-total iron ratio during reaction, dimensionless

In the case of 50% pyrite removal this express reduces to:

$$K_L = \frac{1}{W_p^\circ \ t \ Y^{-2}} \qquad (2)$$

Figure 91 represents a plot of the decrease in S_p ($W_p = 1.88 \ S_p$) as a function of reaction time at 102°C. The rate constant, K_L, for the reaction was determined to be 0.08 W_p^{-1} hr^{-1} for using the relation in Equation (2) above. Earlier studies(3) on a TVA furnished coal revealed a rate constant, K_L, equal to 0.14 W_p^{-1} hr^{-1} for a lower ash (15.5%) starting 1.3 specific gravity sink coal.

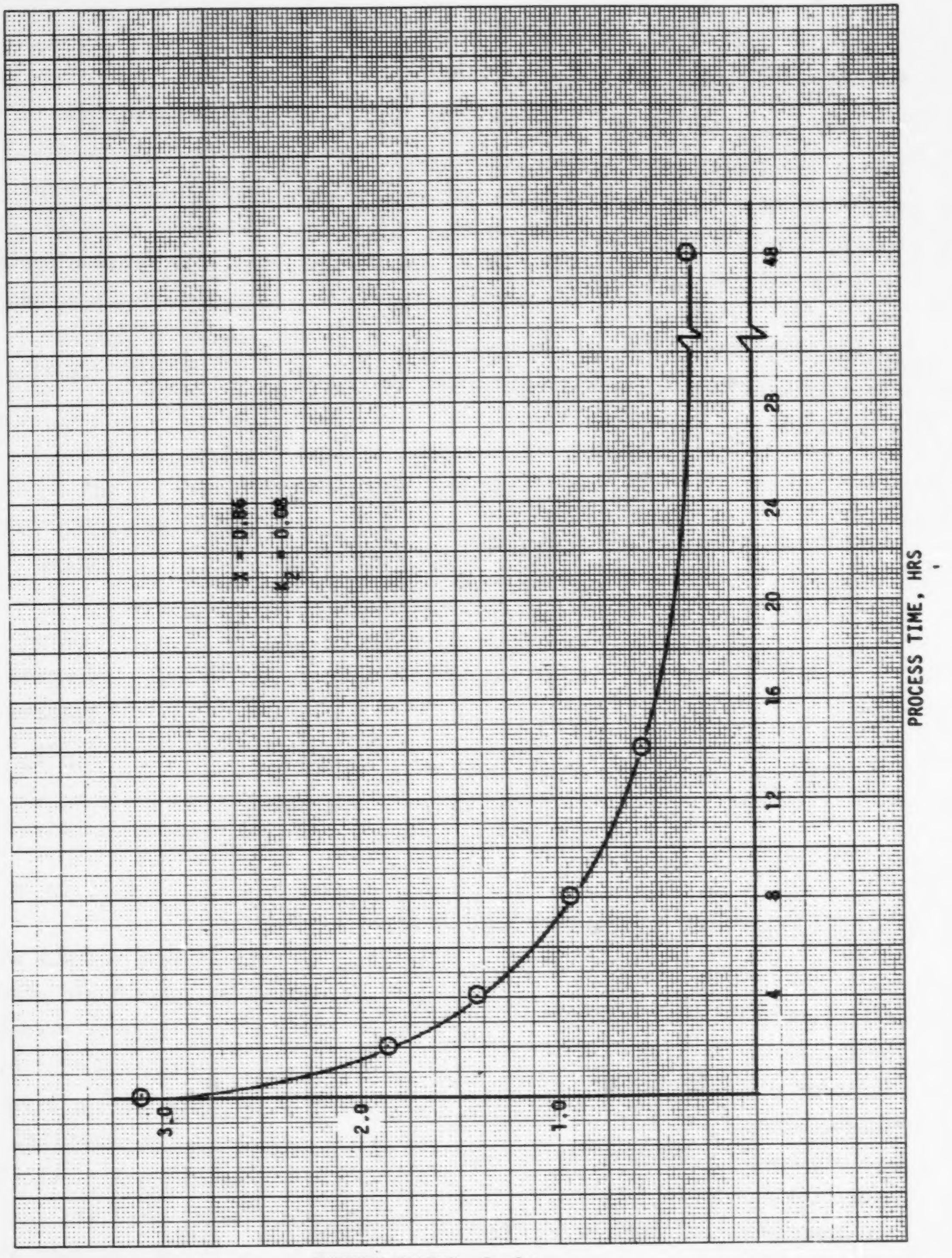


Figure 91. Pyritic Sulfur Leaching Data from 1.3 S.G. TVA Sink Coal (102°C Ambient Pressure).

5.2 GOB PILE COALS (TASK 2A2)

Gob pile samples representing five coal piles were obtained from Banner Industries and Peabody Coal Company. Each of these coals were subjected to float-sink techniques in iron sulfate leach solution and/or conventional organic solvent at various top-sizes. It should be noted that the coal samples were mainly the fine refuse of physical coal-cleaning processing. Only one gob pile (Peabody No. 2) could be gravity separated and/or processed at the 3/8" x 0 particle size, in addition to the as-received size, since the remaining four coal samples were < 1/8" top-size when received (Table 9).

TABLE 9. GOB PILE COAL SAMPLES

Supplier	Coal Designation	Received Coal Top-Size	State
Banner Industries	Enoco	∿ 14 Mesh	Ohio
	J. Role	∿ 14 Mesh	Indiana
	Plymouth	∿ 14 Mesh	Unknown
Peabody Coal Company	Peabody #1	∿ 14 Mesh	W. Ky.
	Peabody #2	+ 1 Inch	W. Ky.

5.2.1 Float Sink - Gob Coals (Task 2A2)

Two gob coals (Table 10) contained large amounts of iron sulfate which dissolved into the separation medium, probably due to the high ionic strength of the solution. The presence of these amounts of iron sulfate is characteristic of weathered coal as often found in gob piles. The lower-sulfur/high heat-content float coal obtained in 46% and 32% yield could provide a very valuable coal product while the sink is probably a throwaway. However, the heat content and total sulfur of the sink Enoco refuse coal is similar to a western coal, and so it might become a product also. Work was discontinued on these two coals at the request of the project officer since the coals were not considered representative of gob pile coal of interest.

TABLE 10. GRAVI-SEPARATION OF BANNER GOB COAL

Coal	% Wt	Dry Basis				$\frac{\text{lbs } SO_2}{10^6 \text{ Btu}}$	
		% Ash	% Sulfur		Btu		
			Total	Sulfate			
Jim Role Raw	100	33.79	3.22	0.75	9171	7.02	
Jim Role Float*	46	8.68	2.33	0.88	12922	3.61	6.56 Wt. Ave.
Jim Role Sink*	54	58.25	2.43	0.21	5352	9.08	
Enoco Raw	100	24.88	3.23	2.00	9556	6.76	
Enoco Float**	32	3.25	2.23	0.06	13630	3.27	2.88 Wt. Ave.
Enoco Sink**	68	28.48	1.30	0.28	9642	2.70	

*Separated in 1.37 sp. gr. iron sulfate solution, roughly 1/8" x 0.

**Separated in 1.33 sp. gr. iron sulfate solution, roughly 1/8" x 0.

Extensive laboratory work was accomplished on the remaining three gob piles as per the statement of work with the exception that float/sink, etc. was only accomplished on 3/8" x 0 Peabody No. 2 coal as noted above.

5.2.2. Float-Sink Analysis (Task 2A2)

Plymouth, Peabody No. 1 and Peabody No. 2 gob coals were gravity separated as described earlier in 1.3 specific gravity iron sulfate leach solution and organic liquid. Table 11 presents the coal analyses of whole coal as well as float-sink fractions of Peabody No. 1 and Plymouth gob pile coals. It is seen that the Plymouth coal is substantially a "clean" coal when compared to the Peabody No. 1 coal. Essentially no difference in total sulfur is noted in the float and sink fractions of the Plymouth coal, although the distribution of sulfur forms is characterized by a higher organic and lower pyritic sulfur in the float fraction as expected.

Peabody No. 1 coal quality is greatly enhanced as a result on gravity separation in Meyers Process leach solution. Particularly noted is the low ash yield obtained with leach solution float fraction (6.2%) as compared to the organic liquid separation (11.8). Likewise a coal product (3.93 lbs $SO_2/10^6$ Btu) is also obtained which meets the Kentucky state standards for allowable SO_2 stack gas emissions (4.0 lbs $SO_2/10^6$ Btu).

Gravity separation of Peabody No. 2 coal (as received, + 1 inch) was likewise accomplished in 1.3 specific gravity iron sulfate leach solution and organic liquid. Table 12 presents the coal analyses of the float/sink fractions.

Gravity separation (Series 1) produced substantially lower yields for the float fractions when compared to previous (Table 7) gravity separations of Peabody coal at ∿ 14 mesh top-size. This is not unexpected since standard gravity separation techniques used at the mine should be more effective in removing "clean" coal at large particle size.

Following the above separation a portion of the sink material was size-reduced to 3/8 inch top-size and subjected to float/sink procedures

TABLE 11. SETTLING TANK FLOAT/SINK SEPARATION/ANALYSES OF GOB PILE COALS (14 MESH)

Gob Pile	Separation Medium @ 20°C	Fraction	Yield %	Ash %, w/w	Heat Content, Btu	Sulfur Content, % w/w				$\frac{\text{lbs } SO_2}{\text{MM Btu}}$
						Total	Pyrite	Sulfate	Organic	
Peabody	--	Whole Coal	--	39.6	8387	3.94	2.30	0.01	1.63	9.39
	--	Whole Coal	--	39.2	8413	3.99	2.56	0.01	1.42	9.49
	Organic* Liquid (1.3 S.G.)	Float	20	11.8	12478	2.76	0.99	0.02	1.75	4.42
		Sink	30	49.9	6814	4.15	3.41	0.07	0.67	12.18
	Leach** Solution (1.3 S.G.)	Float	29	6.2	13419	2.63	0.80	0.02	1.81	3.93
		Sink	71	52.4	6254	4.99	3.45	0.77	0.77	15.96
Plymouth	--	Whole Coal	--	13.6	12495	0.86	0.21	0.01	0.65	1.38
	--	Whole Coal	--	14.6	12815	0.89	0.24	0.00	0.64	1.39
	Organic* Liquid (1.3 S.G.)	Float	39	4.9	14229	0.85	0.13	0.01	0.71	1.19
		Sink	61	20.5	11902	0.91	0.31	0.01	0.59	1.53
	Leach** Solution (1.3 S.G.)	Float	49	4.5	14465	0.92	0.10	0.01	0.81	1.27
		Sink	51	21.7	11790	0.88	0.30	0.02	0.56	1.49

---

[*] Mixture of toluene and perchloroethylene, wt ratio 1:1.35.

[**] Aqueous ferric sulfate (ferri-floc) with specific gravity obtained as follows:

Fe^{+++} as $Fe_2(SO_4)_3$ (wt %)	H_2SO_4 (wt %)	H_2O and $SO_4^{=}$ as $Fe_2(SO_4)_3$ (wt %)
~ 7.5	~ 4.0	~ 88.5

TABLE 12. SETTLING TANK FLOAT/SINK SEPARATION/ANALYSES OF GOB PILE COALS PEABODY #2 COAL (AS-RECEIVED)

Series	Separation Medium @ 20°C	Fraction	Yield %	Ash %, w/w	Heat Content, Btu	Sulfur Content, % w/w				lbs SO_2 / MM Btu
						Total	Pyrite	Sulfate	Organic	
1 (As Received)	Organic* Liquid (1.3 S.G.)	Float	8	10.73	12643	2.92	1.23	0.02	1.67	4.6
		Sink	92	76.16	2623	5.70	5.18	0.02	0.51	43.5
	Leach** Solution (1.3 S.G.)	Float	6	-	-	-	-	-	-	-
		Sink	94	83.50	1002	5.55	5.04	0.29	0.22	110
	--	Starting Coal***		83.50	1002	5.55	5.04	0.29	0.22	110
2 (Ground to 3/8" top-size)	Organic* Liquid (1.3 S.G.)	Float	10	6.23	14071	2.98	1.05	0.01	1.92	4.24
		Sink	90	84.77	1272	5.39	5.20	0.03	0.16	84.8
	Leach** Solution (1.3 S.G.)	Float	9	9.19	13001	4.11	2.25	0.02	1.85	6.3
		Sink	91	85.19	1222	6.26	4.73	0.36	1.18	102

[*] Mixture of toluene and perchloroethylene, wt ratio 1:1.35.

[**] Aqueous ferric sulfate (ferri-floc) with specific gravity obtained as follows:

Fe^{+++} as $Fe_2(SO_4)_3$ (wt %)	H_2SO_4 (wt %)	H_2O and $SO_4^=$ as $Fe_2(SO_4)_3$ (wt %)
~ 7.5	~ 4.0	~ 88.5

[***] Sink portion (above) ground to 3/8" top-size.

again. It is seen in Table 12 (Series 2) that additional float coal is released providing an effective 15% cumulative float recovery from the Peabody No. 2 coal. Thus size reduction to 3/8 inch top-size does increase the yield. However, the 15% cumulative float is still less than the 29% yield observed for 14 mesh Peabody No. 1 coal in Table 11. Further reduction of the sink fraction to -28 mesh did not materially provide additional float product.

5.2.3 Meyers Processing (Task 2A2)

Meyers Process desulfurization was performed on four sink product coals from the foregoing gob coal gravity separations in iron sulfate leach solution. Forty-eight hour processing, at 102°C, of the sink fractions obtained from Plymouth, Peabody No. 1 and Peabody No. 2 gravity separations plus the crushed sink fraction (-28 mesh) from Peabody No. 2 was completed.

Sink coal, obtained from float-sink separation in iron sulfate leach solution of 14 mesh waste coal from Plymouth and Peabody mines, was reacted (Table 13) under Meyers Process conditions for chemical removal of pyritic sulfur. The two steps of float-sink separation in leach solution and Meyers Process treatment of the sink fraction constitute the Gravichem Process.

The Plymouth sink coal containing 22% ash and 1.5 lbs $SO_2/10^6$ Btu before chemical reaction, was reduced to 19% ash and 1.1 lbs $SO_2/10^6$ Btu after five hours reaction at 102°C. The pyritic sulfur content was reduced by 63% (0.19% w/w) with no increase in sulfate or organic sulfur. Continuation of the extraction for as long as 48 hours resulted in no further reduction in sulfur content per unit heat content.

The Peabody coal, containing 52% ash and 16 lbs $SO_2/10^6$ Btu, was reduced to 47% ash and 10 lbs $SO_2/10^6$ Btu after five hours of processing. The pyrite content was reduced by 50% (1.73% w/w) while there was essentially no increase in sulfate and a slight increase in organic sulfur.

TABLE 13. PROCESSING OF GOB PILES SINK FRACTION* @ 102°C (AMBIENT PRESSURE)

Coal	Reaction Time, hrs	Ash %, w/w	Heat Content Btu/lb	Sulfur Content, %, w/w				lbs SO_2 / MM Btu
				Total	Pyrite	Sulfate	Organic	
Plymouth	Control Sink Coal	21.7	11790	0.88	0.30	0.02	0.56	1.49
	5	19.3	12000	0.66	0.11	0.01	0.54	1.10
	24	18.4	12034	0.66	0.07	0.01	0.58	1.10
	48	17.7	12251	0.74	0.07	0.01	0.66	1.21
Peabody #1	Control Sink Coal	52.4	6254	4.99	3.45	0.77	0.77	15.96
	5	46.7	7009	3.46	1.72	0.79	0.95	9.84
	24	49.3	6632	3.89	1.48	1.12	1.29	11.70
	48	42.3	7360	3.46	0.64	1.49	1.33	9.40

*Sink product from 1.3 S.G. settling tank float/sink in leach solution.

Processing up to 48 hours resulted in an 81% reduction in pyritic sulfur but there was irreversible sorption of sulfate and an increase in apparent organic sulfur (probably additional acid insoluble sulfate) with no net reduction in lbs $SO_2/10^6$ Btu.

It can be concluded that both sink coals contained carbonates and clay which tended to irreversibly sorb sulfate from the leach solution on prolonged (greater than 5 hours) treatment allowing no net decrease in sulfur content per unit heat content. This effect was most pronounced for the higher ash Peabody sink coal.

Sink coal obtained from float/sink separation of +1 inch Peabody No. 2 coal in iron sulfate leach solution was reacted at +1 inch top size and at -28 mesh. The -28 mesh coal was obtained by reducing the dried product sink coal (above) to 3/8" top size with subsequent reduction to -28 mesh as described earlier (Section 2.A.1.d). Table 14 presents the results of Meyers Process pyritic sulfur removal from both coals (+1 inch and -28 mesh) after 48 hours at 102°C.

It is seen that processing the sink coal achieves no useful purpose either at +1 inch top size size or -28 mesh. Sulfate retention is high as was noted for Peabody No. 1 coal (Table 13). Basically, however, the high ash content negates useful recovery of this coal.

TABLE 14. PROCESSING OF PEABODY #2 SINK FRACTION* @ 102°C (AMBIENT PRESSURE)

Coal	Reaction Time, hrs	Ash %, w/w	Heat Content, Btu/lb	Sulfur Content, % w/w				$\frac{\text{lbs } SO_2}{\text{MM Btu}}$
				Total	Pyrite	Sulfate	Organic	
+ 1 inch Top-Size	Control sink coal	83.50	1002	5.55	5.04	0.29	0.22	111
	48	81.15	1072	7.77	3.60	3.67	0.50	145
-28 Mesh	Control sink coal	81.79	1044	8.94	5.30	3.18	0.46	171
	48	79.83	717	6.30	0.90	5.37	0.03	176

*Sink product from 1.3 S.G. settling tank float/sink in leach solution.

6.0 SPECIFICATIONS AND ESTIMATES FOR R-1 REACTOR REPLACEMENT

The specifications and a drawing for a replacement desulfurization reactor (R-1) constructed of titanium were sent to four vessel fabricators for price and delivery schedule quotations. The reactor specifications and drawing are presented in Figure 92 and Tables 15 and 16. The following fabricators were contacted for quotations.

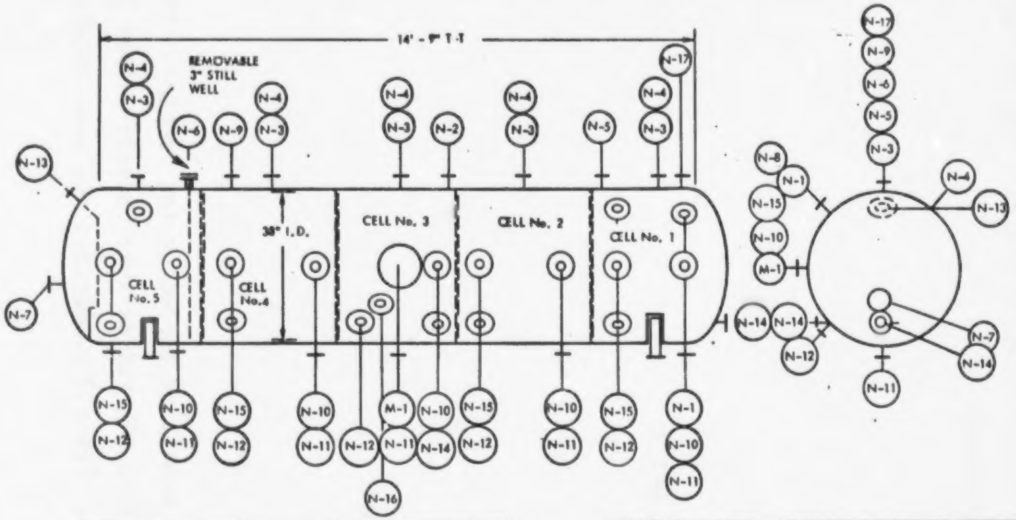
Alloy Specialities, Ltd.
7592 Park Avenue
Garden Grove, CA 92641
(213) 628-6202
Attention: R. H. Higgs

Futura Titanium Corporation
P.O. Box 5004
West Lake Village, California 91359
(213) 873-6912
Attention: Jeff Thomas

Nooter Corporation
P.O. Box 451
St. Louis, Mo 63166
(314) 621-6000
Attention: Gene Smith

Titanium Industries Corporation
17 Industrial Road
Fairfield, New Jersey 07006
(201) 227-5300
Attention: D. Williams

Three of the four fabricators submitted quotes on the reactor. The price ranges were from $59,000 to $115,000 and delivery schedule from 20 to 28 weeks after receipt of order. Table 17 summarizes the bids received.



14' - 9" T-T
REMOVABLE
3" STILL
WELL
38" I.D.
CELL No. 5
CELL No. 4
CELL No. 3
CELL No. 2
CELL No. 1
N-1
N-2
N-3
N-4
N-5
N-6
N-7
N-8
N-9
N-10
N-11
N-12
N-13
N-14
N-15
N-16
N-17
M-1

GASKETS	ASBESTOS COMPOSITION		1/16 IN.
BOLTS	C.S.		
INTERNALS	TI		*
NOZZLES	TI		*
VESSEL SUPPTS.	CARBON STEEL	SA-36	*
TRAYS			
LINING			
HEADS	TI		*
SHELL	TI		*
ITEM	MATERIAL	ASTM	THICK

* BY VENDOR

DESIGN PRESSURE: INTERNAL 150 PSIG; EXTERNAL 14,7 PSI AT 300 °F.
OPERATING PRESSURE 110 PSIG; TEMPERATURE 265 °F.

N-17	1	1 IN	150 #	R F	PRESS. SW.
M-1	1	16 IN	150 #	RF	MANWAY
N-16	1	1-1/2 IN	↓	↓	THERMOWELL
N-15	4	1-1/2 IN			THERMOWELL
N-14	2	2 IN			STEAM INLET
N-13	1	2 IN			OUTLET
N-12	5	1-1/2 IN			PUMP RECYCLE
N-11	5	1-1/2 IN			PUMP SUCTION
N-10	5	1 IN			SAMPLE
N-9	1	2 IN			VAPOR OUTLET
N-8	1	1 IN			PRESS. GAGE
N-7	1	3 IN			LEVEL SWITCH
N-6	1	4 IN			LEVEL CONTROL
N-5	1	2 IN			RELIEF VALVE
N-4	5	6 IN			RUPTURE DISK
N-3	5	12 IN			MIXER
N-2	1	2 IN			CELL No. 3 INLET
N-1	1	2 IN			CELL No. 1 INLET
MARK	NO. REQ.	SIZE	RATING	FACING	SERVICE
			NOZZLES		

8/23/78

TRW
SYSTEMS GROUP
ONE SPACE PARK • [illegible] CALIFORNIA

PRIMARY REACTOR
R-1

Figure 92. Reactor Drawing.

TABLE 15. DESIGN NOTES

1. Vessel to be constructed according to Unifered Pressure Vessel Code, Section VIII, Div. 1. Code stamp required.
2. Vessel contains coal slurry (Sp.Gr. 1.30). Slurry consists of 20-40% coal particle (8 mesh top-size) in liquid. Liquid is 4% H_2SO_4, 3% $FeSO_4$, 22% $Fe_2(SO_4)_3$ in water.
3. Corrosion allowance: Shell 1/16"
   Heads 1/16"
   Nozzles 1/16"
   Internals 1/16" per side
4. X-Ray - Yes. Stress Relief - No.
5. Design loads per Uniform Building Code. Basic wind load - 20 psf. Design cosmic coeff. - UBC Zone 3.
6. Internals furnished and installed by vendor.
7. Manhole cover should be hinged.
8. Fireproofing not required. Insulation by purchaser.
9. Paint C.S. surfaces one shop coal red oxide primer (1-1/2 mil D.F.T.) Surface preparation - SSPC-SP-2003.
10. Live loading due to mixers
11. Design based on 2:1 S.E. heads. Substitution permitted only if liquid volume remains unchanged.
12. Internal baffles shall be removable by manway. Detail design by vendor

TABLE 16. FABRICATION NOTES

1. Flange bolt holes shall straddle normal vessel centerlines.
2. Shell and head seams shall equal double welded butt joint where possible.
3. Nozzles and couplings shall be full penetration welded.
4. Each nozzle and manhole reinforcing pad or segment thereof shall have a 1/8" NPT test hole located 90° off the vessel centerline.
5. All tray elevations are to top of tray support ring.
6. Nozzles, manholes and trays shall have same designation as shown on attached drawing.
7. Fabricator's shop drawings issued for approval shall indicate 1) shell head, skirt and internals thickness, 2) ASTM numbers for all materials, 3) head and shell sear joint efficiency, 4) name-plate layout, 5) total weight empty (with and without trays) and full of water, and 6) support details.
8. Nameplate shall be stainless steel and preferably located adjacent to lower manhole. Support brackets shall be used on insulated vessels.
9. Vessel shall be cleaned inside and outside of all dirt, grease scale and debris.
10. Prior to shipping all openings shall be protected with covers or thread protectors.

TABLE 17. REACTOR BID SUMMARY

R-1 Desulfurization Reactor - 38" ø x 14'9" T/T, Ti, D.P. 150 psig/-14.7 psig @ 300°F

Fabricator	Price	Delivery Schedule	Remarks
Astro Metallurgical Corp. (Alloy Specialities)	$ 58,925 FOB	20-24 weeks after receipt of approved drawings	Wall thickness - .3199 in. min Head thickness - .3172 in. min
Futura Titanium Corp.	$ 66,000 FOB	18-20 weeks after receipt of approved drawings	Wall thickness - 3/8"
Nooter Corp.	$115,000 FOB	24 weeks after receipt of order	Wall thickness - 7/16 in. Head thickness - .38 in. min

7.0 RTU ACETONE EXTRACTION UNIT

This section of the report documents the results of an RTU downstream processing unit conceptual design study. The effort was aimed at defining the following: (1) those operations which are required to extract the elemental sulfur from the RTU processed coal cake; (2) the appropriate commercially available equipment which can be integrated into a downstream processing unit (to be referred to henceforth as the Tail-End Unit, TEU); and (3) an estimated cost to design and construct the envisioned RTU addition.

Section 7.1 contains a brief discussion of the conceptual TEU, including such items as a process description, TEU flow diagram, mass balance, utility requirements equipment design basis, plot plan and elevation sketch. Section 7.2 presents an equipment list, estimated equipment costs, and an estimate of total TEU erected plant cost.

7.1 TEU CONCEPTUAL DESIGN

The TEU conceptual design has as its foundation several years of previously accomplished bench-scale sulfur extraction studies and accumulated laboratory data(1-4). Also significant insight into the design of the TEU was gained during previously completed conceptual full-scale Meyers Process and Gravichem Process design activities(1-5) as well as a prior Meyers Process pilot plant design study(6).

The background knowledge and information lead to a TEU design which incorporates RTU generated wet coal cake handling and transport, solvent contacting sulfur-rich solvent extraction from the coal cake, coke drying with solvent recovery, solvent purification with sulfur recovery, waste water neutralization, and solvent storage operations. The integrated unit, as designed, is sized to be compatible with RTU operations in a continuous mode. A flow diagram of the TEU is presented as Figure 93. The diagram presents equipment specifications as well as flow patterns and identifying stream numbers. The corresponding mass balance volumetric flows, and explanatory notes are shown in Table 18. The system steam, cooling water, process water and electrical requirements are tabulated in Table 19. A conceptual level TEU pilot plan and front elevation are shown in Figures 94 and 95 respectively while a perspective sketch of TEU may be seen as Figure 96.

7.1.1 Process Description

The following paragraphs present a brief discussion of each of the major subsystems of the TEU.

7.1.1.1 Acetone/Coal Contacting System

Coal which has been processed in the RTU and stored in tote bins is fed to the coal feed bin (TX-3) which has a capacity for 10 tons of coal. The feed bin is blanketed with nitrogen and vented to the vent gas system to allow for acetone wet coal to be recycled to the contacting system. The bin discharger (DRX-1) maintains a steady rate of coal up to 500 lb/hr to the contactor (VX-1). The coal feed bin will hold enough coal for about a 40 hour run to the contacting system. The contactor (VX-1) is a three stage horizontal vessel with a mixer in each stage. The contactor is sized to provide one hour residence time with a 500 lb/hr coal rate and a 1000 lb/hr acetone rate. The vessel has adjustable weirs to allow for changes in residence time by adjusting the volume from 1/3 to 2/3 full. The acetone feed is supplied from underground storage tank (TX-1) and pump PX-1. The feed acetone is heated to 133°F in exchanger EX-1 and the contactor slurry is maintained at 133°F by a pump-around loop using pump PX-2 and exchanger EX-2. The coal slurry from the contactor is pumped (PX-3) to the centrifuge (SX-1). The centrifuge is sized for 500 lb/hr coal and 1100 lb/hr acetone/water producing a coal cake containing about 15% acetone. The coal cake from the centrifuge can be fed to the drying system or stored in tote bins for further acetone contacting if necessary. The centrate from SX-1 is pumped to the distillation feed storage (TX-2).

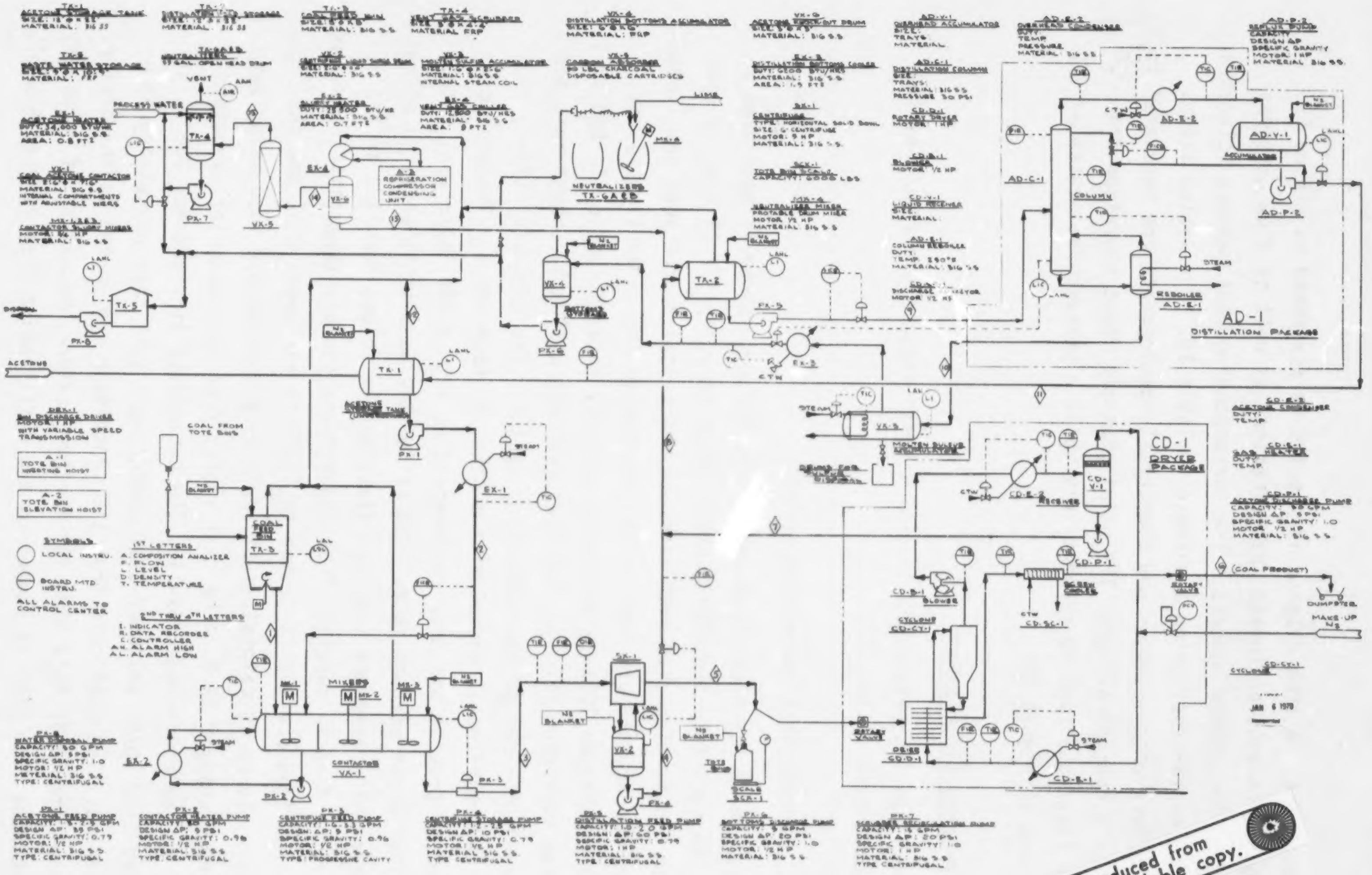

AD-1
DISTILLATION PACKAGE
CD-1
DRYER PACKAGE
ACETONE
COAL FROM TOTE BINS
PROCESS WATER
VENT
LIME
NEUTRALIZERS TK-6 A&B
REFRIGERATION COMPRESSOR CONDENSING UNIT
CONTACTOR
VA-1
MIXERS
COLUMN
AD-C-1
ACCUMULATOR
AD-V-1
AD-P-2
AD-E-2
REBOILER
AD-E-1
STEAM
CTW
TA-1
TA-2
TA-3
TA-4
TA-5
VK-5
VK-2
VK-3
VK-6
SK-1
EX-1
EX-2
EX-3
EX-4
PX-1
PX-2
PX-3
PX-4
PX-5
PX-6
PX-7
PX-8
N2 BLANKET
DISPOSAL
CD-V-1
CD-P-1
CD-E-2
RECEIVER
CD-E-1
CD-D-1
CD-B-1
BLOWER
CYCLONE
CD-CY-1
SCREEN
CD-SC-1
ROTARY VALVE
(COAL PRODUCT)
DUMPSTER
MAKE-UP N2
SCALE
SCA-1
TOTE BIN
A-1
TOTE BIN WEIGHING HOIST
A-2
TOTE BIN ELEVATION HOIST
SYMBOLS
LOCAL INSTRU.
BOARD MTD. INSTRU.
ALL ALARMS TO CONTROL CENTER
1ST LETTERS
A. COMPOSITION ANALIZER
F. FLOW
L. LEVEL
D. DENSITY
T. TEMPERATURE
2ND THRU 4TH LETTERS
I. INDICATOR
R. DATA RECORDER
C. CONTROLLER
AH. ALARM HIGH
AL. ALARM LOW
JAN 6 1970

Figure 93. Process Flow Diagram.

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TABLE 18. ACETONE EXTRACTION
PRINCIPAL MASS AND VOLUMETRIC FLOWS

Stream Number & Description	Pounds/Hour				GPM	Density lb/ft^3	Pressure psig	Temp. ^{o}F
	Coal	Water	Acetone	Total				
1. Coal to Contactor	500	33(1)	0	533	-	81	0	70
2. Acetone to Contactor	0	0	1000	1000	2.5	49	10	133
3. Acetone/Coal Slurry ex. Contractor	500	33	1000	1533	3.3	60	2	133
4. Centrate ex. Centrifuge	0	30(2)	915	945	2.5	49	2	133
5. Coal ex. Centrifuge	500	3	85	588	-	80	2	130
6. Dried Coal ex. Dryer Package	500	0	0	500	-	81	0	
7. Acetone/Water ex. Dryer Package	0	3	85	88	0.2	49	2	
8. Spent Acetone to Storage	0	33	1000	1033	2.6	49	2	120
9. Distillation Feed (2)	0	25	765	790	2.0	49	30	70
10. Distillation Bottoms (3) (4)	0	25	0	25	0.1	62	30	250
11. Redistilled Acetone (3)	0	0	765	765	1.9	49	30	100

(Continued)

TABLE 18. (Continued)

Stream Number & Description	Pounds/Hour Nitrogen	Pounds/Hour Water	Pounds/Hour Acetone	Pounds/Hour Total	GPM	Density lb/ft^3	Pressure psig	Temp. °F
12. Vapor displaced by filling Acetone Storage Tank	47.9	Trace	47.9	95.8(6)	100(6)	-	1	100
13. Acetone recovered from chiller	0	Trace	44.5	44.5	0.1	49	1	0
14. Vapor to Carbon Adsorbers	47.9	0	3.4	51.3	-	-	1	0
15. Vent Gas	47.9	0	Trace	47.9	-	-	1	20

NOTES:

(1) Moisture content on incoming coal will vary from 20% to 0.2% of the weight of dry coal.

(2) Water in centrate varies from 92 lb/hr in first extraction to 1 lb/hr in third extraction.

(3) Distillation Package designed to run at lower feed rates and a higher service factor.

(4) Distillation bottoms will contain 1.5 lbs. of sulfur per hour.

(5) Maximum instantaneous flow rates in vent system.

(6) Assumes acetone charged to TX-1 at 100 GPM and a 50/50 mixture (by weight) of acetone and nitrogen in the vapor space.

TABLE 19. UTILITY REQUIREMENTS

STEAM		
	EX-1	34 lb/hr
	EX-2	26 lb/hr
	Distillation Unit	800 lb/hr
	Dryer Unit	60 lb/hr
	TOTAL	920 lb/hr
COOLING WATER		
	EX-3	1 GPM
	Distillation Unit	70 GPM
	Dryer Unit	6 GPM
	TOTAL	77 GPM
PROCESS WATER		
	TX-4	1 GPM
ELECTRICAL	**CONNECTED LOAD**	**ESTIMATED ENERGY CONSUMPTION**
	90 KVA	65 KW 522 KWH for an 8-Hour Shift.

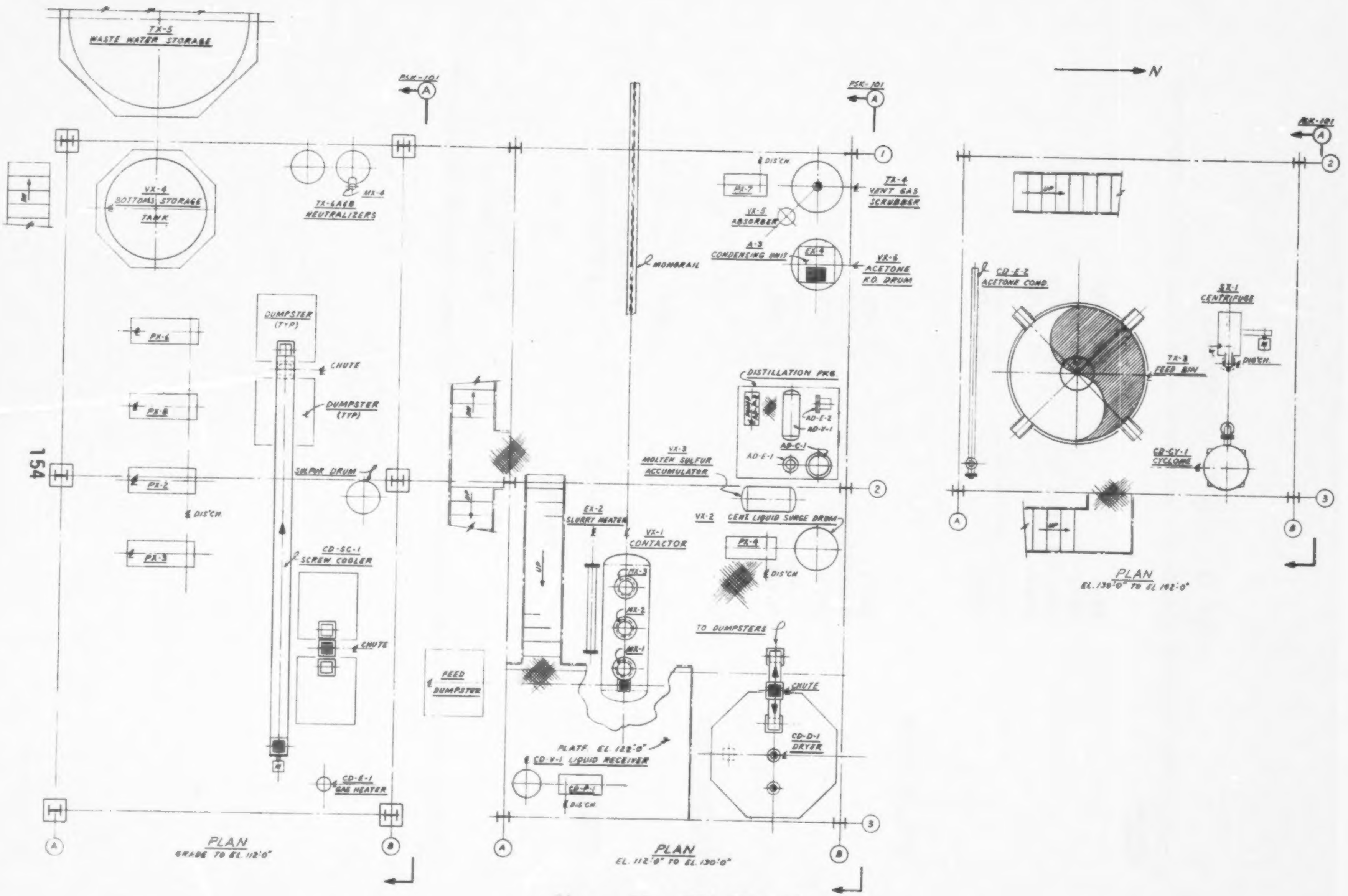
TX-5
WASTE WATER STORAGE
VX-4
BOTTOMS STORAGE
TANK
TX-6A&B
NEUTRALIZERS
MX-4
PSK-101
DUMPSTER
(TYP)
CHUTE
DUMPSTER
(TYP)
PX-6
PX-8
PX-2
PX-3
DIS'CH
SULFUR DRUM
CD-SC-1
SCREW COOLER
CHUTE
FEED
DUMPSTER
CD-E-1
GAS HEATER
PLAN
GRADE TO EL. 112'-0"
MONORAIL
PSK-101
DIS'CH
PX-7
TX-4
VENT GAS
SCRUBBER
VX-5
ABSORBER
A-3
CONDENSING UNIT
EX-4
VX-6
ACETONE
K.O. DRUM
DISTILLATION PKG.
AD-E-2
AD-V-1
AD-C-1
AD-E-1
VX-3
MOLTEN SULFUR
ACCUMULATOR
VX-2
CENT. LIQUID SURGE DRUM
EX-2
SLURRY HEATER
VX-1
CONTACTOR
MX-3
MX-2
MX-1
UP
PX-4
DIS'CH
TO DUMPSTERS
CHUTE
CD-D-1
DRYER
PLATF. EL. 122'-0"
CD-V-1 LIQUID RECEIVER
CD-P-1
DIS'CH
PLAN
EL. 112'-0" TO EL. 130'-0"
N
PSK-101
UP
CD-E-2
ACETONE COND.
TX-3
FEED BIN
SX-1
CENTRIFUGE
DIS'CH.
CD-CY-1
CYCLONE
UP
PLAN
EL. 130'-0" TO EL. 142'-0"
A
B
1
2
3

Figure 94. TEU Plot Plan.

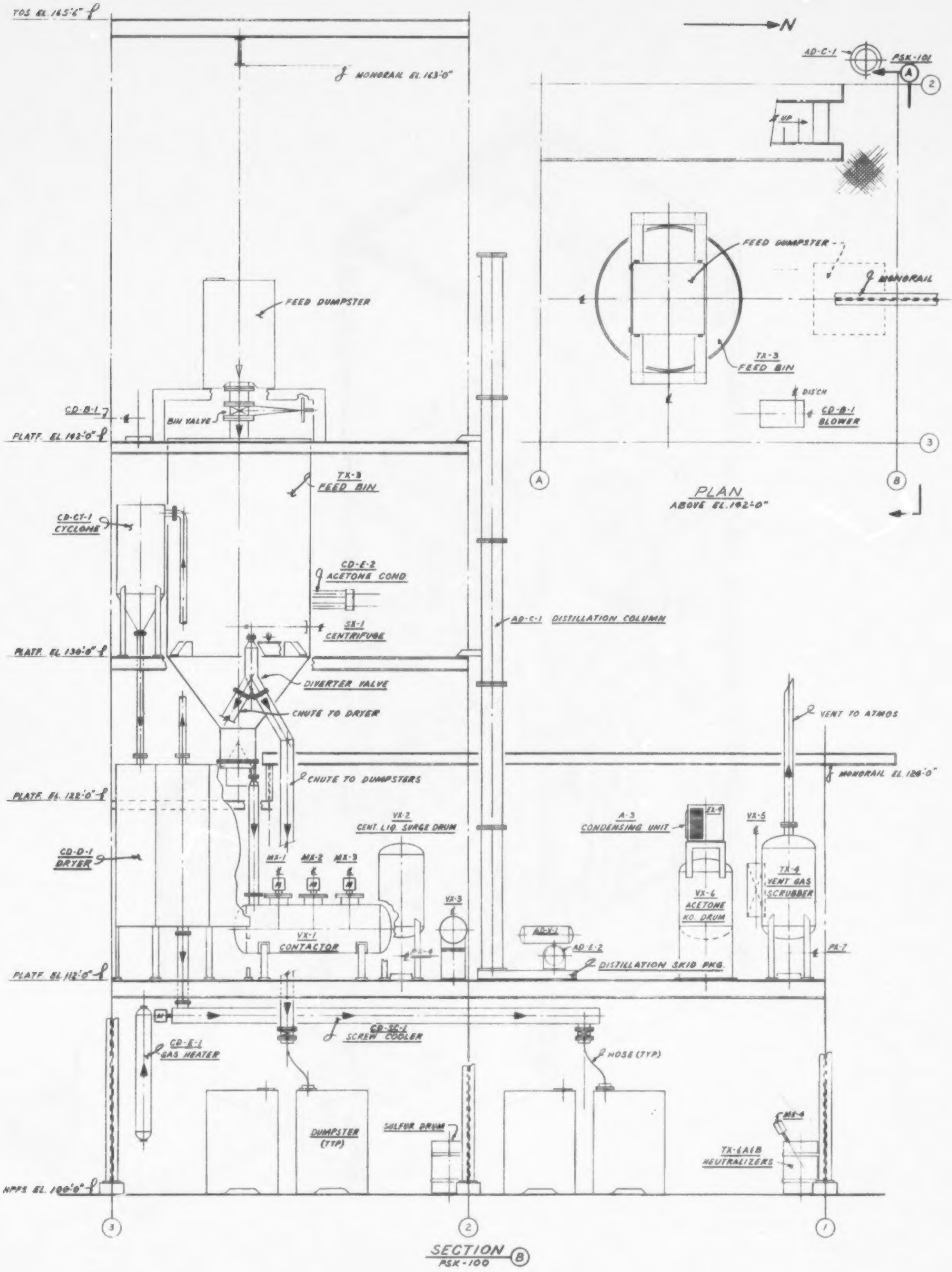
TOS EL 165'-6"
MONORAIL EL 163'-0"
FEED DUMPSTER
CD-B-1
BIN VALVE
PLATF. EL 142'-0"
TX-3
FEED BIN
CD-CY-1
CYCLONE
CD-E-2
ACETONE COND
SX-1
CENTRIFUGE
AD-C-1 DISTILLATION COLUMN
PLATF. EL 130'-0"
DIVERTER VALVE
CHUTE TO DRYER
VENT TO ATMOS
CHUTE TO DUMPSTERS
MONORAIL EL 128'-0"
PLATF. EL 122'-0"
VX-2
CENT. LIQ. SURGE DRUM
A-3
CONDENSING UNIT
EX-9
VX-5
CD-D-1
DRYER
MX-1
MX-2
MX-3
TX-4
VENT GAS
SCRUBBER
VX-6
ACETONE
KO. DRUM
VX-3
VX-1
CONTACTOR
PX-4
AD-V-1
AD-E-2
DISTILLATION SKID PKG.
PX-7
PLATF. EL 112'-0"
CD-SC-1
SCREW COOLER
CD-E-1
GAS HEATER
HOSE (TYP)
DUMPSTER
(TYP)
SULFUR DRUM
MX-4
TX-6A&B
NEUTRALIZERS
HPFS EL 100'-0"
3
2
1
SECTION
PSK-100
B
N
AD-C-1
PSK-101
A
2
UP
FEED DUMPSTER
MONORAIL
TX-3
FEED BIN
DIS'CH
CD-B-1
BLOWER
3
A
B
PLAN
ABOVE EL. 142'-0"

Figure 95. TEU Front Elevation.

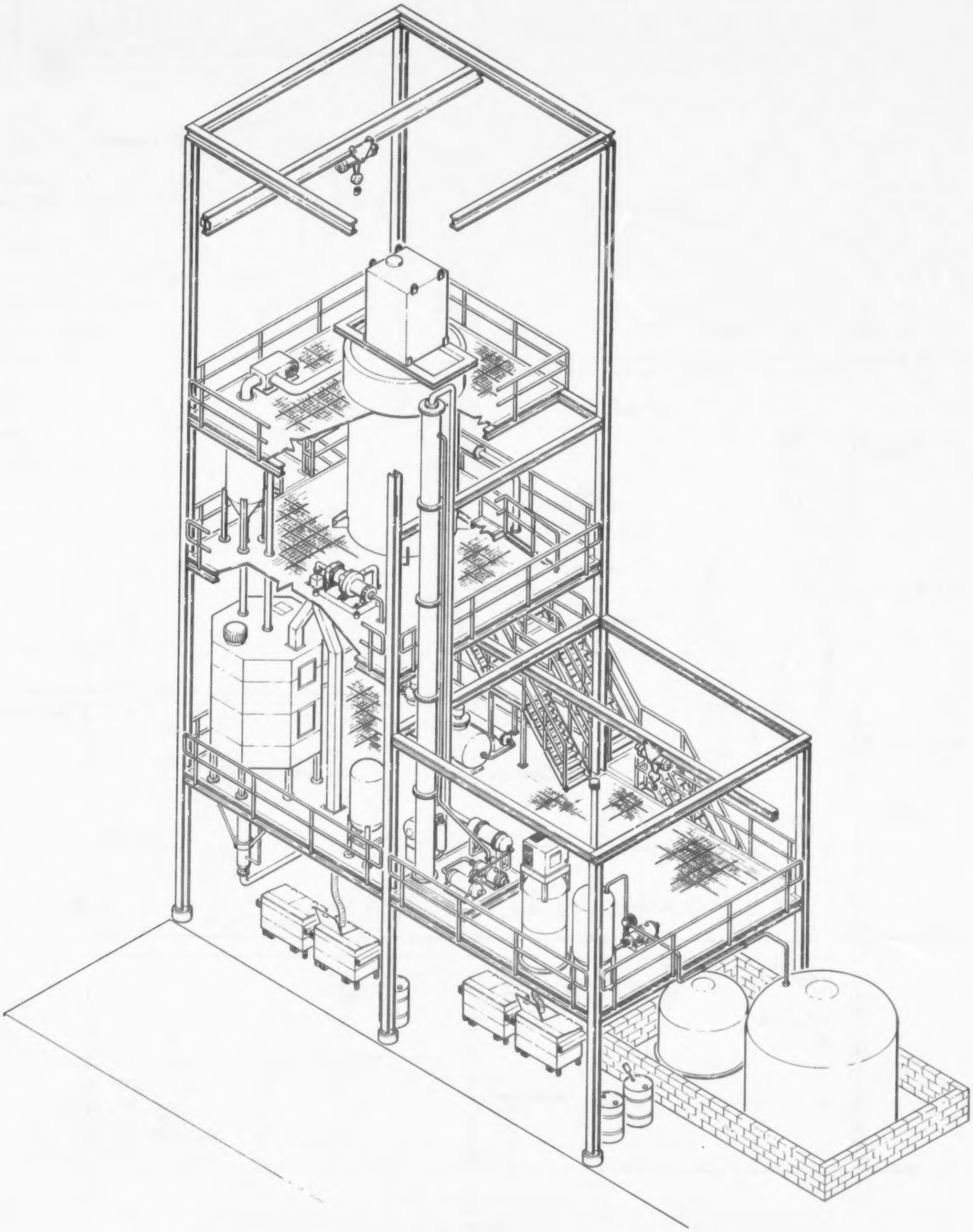

Figure 96. Tail-End Unit Sketch.

7.1.1.2 Coal Drying System

The coal drying system is sized for 500 lb/hr coal containing 15 wt. % volatiles and drying to a coal product containing 0.5 wt. % volatiles. The dryer is a closed loop system utilizing inert gas as the drying media. The coal is fed to the dryer from the centrifuge where it is contacted with a hot inert gas stream and dried at 225°F. The dried coal is cooled and conveyed into dumpsters via the screw cooler (CD-SC-1). The inert gas off the dryer containing acetone and water passes through the cyclone separator (CD-CY-1), the recycle gas blower (CD-B-1), condenser (CD-E-2) and the condensate receiver (CD-V-1). The acetone condensate is pumped to the distillation feed storage tank (TX-2) and the gas is recirculated to the dryer after heating to 400°F in the recycle gas heater (CD-E-1).

7.1.1.3 Acetone Recovery System

The distillation feed storage tank (TX-2) is sized to accumulate enough acetone/water mixture to allow for one week of continuous operation of the contacting system at the 500 lb/hr coal rate. The acetone distillation system is designed for a feed rate of 2 gpm acetone/water mixture with the overhead product to be 98.5% acetone. The column which is 14 inch diameter with 30 trays will operate at 30 psig such that the sulfur in the bottoms will be molten. The acetone overhead product is pumped to the underground acetone storage tank (TX-1) which is sized to contain enough acetone for a seven day run at design rates. The bottoms product containing water and molten sulfur is pressured to the molten sulfur accumulator (VX-3) where the sulfur phase is decanted into drums for disposal. The molten sulfur accumulator is designed to hold one week's production of sulfur at the design rate. The water phase from the molten sulfur accumulator is cooled in exchanger EX-3 and stored in the bottoms storage VX-4 which is sized to hold enough material for two weeks operation. The water phase material in the bottoms storage is pumped to the neutralizers (TX-6A & B) where the material is neutralized with lime or held in the waste water storage (TX-5) for disposal. All of the processing equipment in acetone service is blanketed with nitrogen and vented to a common acetone scrubbing system. The scrubbing system consists of a chilled condenser

(EX-4), acetone K.O. drum (VX-6), a carbon bed adsorber (VX-5) which contains disposable cartridges, and a recirculated water scrubber. The condensed acetone is returned to the distillation feed storage for reuse. The spent scrubber water is pumped to the waste water storage tank (TX-5) and held for disposal.

7.1.2 TEU Design Basis

The overall TEU conceptual design is based on a processing unit capable of handling a nominal throughput of 500 lbs/hr of coal sized up to 8 mesh top-size. The plant is designed to operate on a continuous basis with sufficient liquid storage capacity to allow for up to 7 days of uninterrupted run duration. The specific design basis for each piece of major process equipment is presented in Table 20.

7.2 TEU COST ESTIMATE

An estimate of the cost to fully engineer, specify, procure and construct the TEU adjacent to the RTU at TRW's Capistrano Test Site was completed. The overall cost estimate is generally of a factored nature based primarily on FOB major equipment costs. The equipment list and associated equipment costs (FOB) are presented in Table 21. As may be determined from the list, the total FOB major equipment cost is estimated to be $387,200 (mid 1978 basis). A complete construction cost estimate is shown in Table 22. The elements of the total estimate are delineated in Table 22 as is the source of each estimated value. As may be seen, the total estimated cost (escalated to mid 1979) to engineer the TEU, specify and procure all required equipment, and completely construct the unit is $1,727,700.

TABLE 20. TEU EQUIPMENT DESIGN BASIS SUMMARY

Equipment Number	Service	Design Basis
TX-1	Acetone Storage	Sized to contain enough acetone for a 7-day production run using 1000 lbs of acetone per hour to contact the coal in VX-1. This tank will be underground.
TX-2	Distillation Feed Storage	Sized to accumulate enough acetone/water mixture to allow 1 weeks continuous operation at an acetone contacting rate of 1000 lb/hr. This tank will be underground.
TX-3	Coal Feed Bin	Sized to hold 10 tons of coal feed with a coal bulk density of 50 lb/ft^3.
TX-4	Vent Gas Scrubber	Removes any vapors from vent streams prior to exhaust to atmosphere. Operates at atmospheric pressure and uses water at the scrubbing medium.
TX-5	Waste Water Storage	Sized to hold 1/2 weeks waste water from the vent gas scrubber (inlet scrubbing water rate of 1 gpm) and the distillation bottoms stream (33 lb/hr.).
TX-6 A&B	Neutralizers	These are 55 gal, open-head drums for making test and sample quantities of by-product for further evaluation.
VX-1	Coal/Acetone Contactor	Sized for 1 hour residence time using 2/3 of the available volume at maximum (adjustable) wier height. Maximum coal rate is 500 lb/hr and maximum acetone rate is 1000 lb/hr. Minimum wier height is 1/3 the diameter.
VX-2	Centrifuge Liquid Surge	Sized for 1 hour residence time for acetone/water mixture for maximum acetone contactor rate of 1000 lb/hr and 88 lbs/hr acetone on the solid coal product from the centrifuge.

(Continued)

TABLE 20. (Continued)

Equipment Number	Service	Design Basis
VX-3	Molten Sulfur Accumulator	Sized to allow all sulfur from a 1-week run to accumulate in 60% of the volume of the vessel. This assumes a coal rate of 500 lb/hr, a sulfur content of 0.4 wt. percent on the incoming coal, and a 90% sulfur extraction efficiency.
VX-4	Distillation Bottoms Accumulator	Sized to hold the water product for two weeks prior to disposal or neutralization.
VX-5	Carbon Adsorption Panels	These are re-chargeable, filter-type panels containing 50 lbs. of charcoal that will adsorb 8 lbs of acetone before replacement of carbon.
VX-6	Acetone Knock-Out Drum	Sized to allow separation between condensed acetone and vapors vented to atmosphere. This vessel will operate at 0°F.
PX-1	Acetone Feed Pump	Supplies acetone from underground storage to the acetone/coal contactor at a maximum rate of 1000 lb/hr.
PX-2	Contactor Heater Pump	Re-circulates acetone/coal slurry from the first section of the contactor, through a heater and back to the contactor.
PX-3	Centrifuge Feed Pump	Pumps slurry from the last section of the contactor to the centrifuge. This pump will be a variable-speed progressive cavity pump and will operate on level control from the contactor.
PX-4	Centrate Storage Pump	Recovers acetone/water mixture from the centrifuge and sends to underground storage. Assumes acetone/water rate of 1012 lbs/hr. (15% volatiles on solid coal from the centrifuge).
PX-5	Distillation Feed Pump	Sends acetone/water mixture from underground storage to distillation package. Assumes distillation package will run at 2 gpm and 30 psig.

(Continued)

TABLE 20. (Continued)

Equipment Number	Service	Design Basis
PX-6	Bottoms Discharge Pump	Assumes water rate to be 33 lb/hr (4 gph). At a pumping rate of 5 g.p.m., a day's accumulation would be discharged to disposal in 20 minutes.
PX-7	Scrubber Recirculation Pump	Pumps water from the scrubber bottoms to a distributor in the top of TX-4 or to disposal. Enough water must be recycled to mix with make-up water to keep the packing material wet.
PX-8	Water Disposal Pump	Empties contents of TX-5 to disposal in 3 hours.
EX-1	Acetone Heater	Heat acetone from ambient temperature to 133°F. Design duty is 34,000 btu/hr. Heat transfer coefficient is 130 btu/ft^2 hr. °F.
EX-2	Slurry Heater	Provides heat to increase coal temperature from ambient to 133°F and to add heat for any losses to ambient in the contactor. Design duty is 25,500 btu/hr and the heat transfer coefficient is 130 btu/ft.^{2} hr °F.
EX-3	Distillation Bottoms Cooler	Cools bottoms temperature to 100°F for safe storage and handling. Design duty is 6200 btu/hr and the heat transfer coefficient is 60 btu/ft.^{2} hr. °F.
EX-4	Vent Gas Chiller	Cools the vent gas stream to allow greater acetone recovery. Design duty is 12,500 btu/hr and the heat transfer coefficient is 25 btu/ft.^{2} hr. °F.

(Continued)

TABLE 20. (Continued)

Equipment Number	Service	Design Basis
MX-1,2 & 3	Contactor Slurry Mixers	Maintain homogeneous slurry within contactor and promote acetone contact with coal particles.
MX-4	Portable Drum Mixer	Mixes reactants and promotes neutralization reaction.
Scale	Tote Bin Scale	Provides data on tote bin weight during loading. Maximum gross weight assumed 6000 lbs.
SX-1	Slurry Centrifuge	Separate liquid and solids. 500 lbs/hr. coal and 1100 lb/hr. acetone and water. Volatile content of coal product to be 15%.
BDX-1	Bin Discharger	Prevents arching and feeds wet coat to the contactor at a uniform rate.

TABLE 21. EQUIPMENT LIST AND FOB COST*

Item No.		$K, FOB
A-1	Tote Bin Inverting Hoist - 2 ton capacity	4
A-2	Tote Bin Elevation Hoist - 2 ton capacity	4
A-3	Refrigeration Unit - 1 ton	3
AD-1	Distillation Package	80
AD-C-1	Distillation Column - 316 SS	as AD-1
AD-E-1	Column Reboiler - 316 SS	as AD-1
AD-E-2	Overhead Condenser - 316 SS	as AD-1
AD-P-2	Reflux Pump - 1 HP, 316 SS	as AD-1
AD-V-1	Overhead Accumulator - CS	as AD-1
BDX-1	Bin Discharger	9
CD-1	Coal Dryer Package	91
CD-B-1	Recycle Gas Blower	as CD-1
CD-CY-1	Cyclone Separator	as CD-1
CD-D-1	Dryer - 6'6"ø x 10'6", 1 HP, 304 SS	as CD-1
CD-E-1	Recycle Gas Heater	as CD-1
CD-E-2	Acetone Condenser	as CD-1
CD-P-1	Acetone Discharge Pump - 30 GPM, 316 SS, 1-1/2 HP	as CD-1
CD-SC-1	Discharge Conveyor - 1/2 HP	as CD-1
CD-V-1	Liquid Receiver	as CD-1
DRX-1	Bin Discharger Driver - 1 HP, Var. Speed	0.5
EX-1	Acetone Heater - 0.8 ft^2, 316 SS	.3
EX-2	Slurry Heater - 0.7 ft^2, 316 SS	.3
EX-3	Distillation Bottoms Cooler - 1.5 ft^2, 316 SS	.4
EX-4	Vent Gas Chiller - 17 ft^2, 316 SS	2.6

*Mid 1978 basis.

(Continued)

TABLE 21. (Continued)

Item No.		$K, FOB
MX-1,2,&3	Contactor Slurry Mixers - 1/2 HP ea., 316 SS	6.5
MX-4	Neutralizer Mixer - 1/2 HP, 316 SS	.6
PX-1	Acetone Feed Pump - 1.3-2.5 GPM, 1/2 HP, 316 SS	3
PX-2	Contactor Heater Pump - 30 GPM, 1/2 HP, 316 SS	1.4
PX-3	Centrifuge Feed Pump - 1.6-3.1 GPM, 1/2 HP, 316 SS	4.2
PX-4	Centrifuge Storage Pump - 1-3.2 GPM, 1/2 HP, 316 SS	4.2
PX-5	Distillation Feed Pump - 1-3.2 GPM, 1 HP, 316 SS	3
PX-6	Bottoms Discharge Pump - 5 GPM, 1/2 HP, 316 SS	2.5
PX-7	Scrubber Circulation Pump - 15 GPM, 1 HP, 316 SS	1.8
PX-8	Water Disposal Pump - 30 GPM, 1/2 HP, 316 SS	1.8
SX-1	Centrifuge - 6" Solid Bowl, 5 HP, 316 SS	24
SCX-1	Tote Bin Scale - 6000 lb Capacity, Electric	7.5
TX-1	Acetone Storage Tank - 12' ø x 22', FRP	46.7
TX-2	Distillation Feed Storage Tank - 12' ø x 22', FRP	46.7
TX-3	Coal Feed Bin - 8' ø x 8', 316L SS	15
TX-4	Vent Gas Scrubber - 3' ø x 4'4", FRP	1
TX-5	Waste Water Storage - 9' ø x 10'9", FRP	4
TX-6	Neutralizers (2) - 55 Gal, Open Head Drum	-
VX-1	Coal Acetone Contactor - 2'6"ø x 7'6", 316 SS	5
VX-2	Centrifuge Liquid Surge Drum - 2'6"ø x 4', 316 SS	3.8
VX-3	Molten Sulfur Accumulator - 1'6"ø x 2'6", 316 SS	3
VX-4	Distillation Bottoms Accumulator - 6' ø x 6', FRP	2.1
VX-5	Carbon Adsorber - 50 lb Carbon Cartridges	.5
VX-6	Acetone K.O. Drum - 3' ø x 3', 316 SS	3.8

TABLE 22. ESTIMATED TEU CONSTRUCTION COSTS

Element	Basis	Materials	Sub-Contract	Labor	Total
FOB Equipment	TRW Estimate	$387,200			
Site Preparation	"		$ 17,200		
Concrete	"		48,000		
Structural Steel	"		158,300		
Piping	"	140,400		$74,400	
Instrumentation	"	101,600		25,400	
Electrical	"		133,300		
Insulation	5% FOB Equip.		19,400		
Painting	3.8% FOB Equip.		14,700		
Freight	3% Mat'l	18,900			
TOTAL DIRECT COST		648,100	390,900	99,800	$1,138,800
Sales Tax	6% Mat'l (less frt.)				36,600
Const. Tools	20% Labor				20,000
Labor Fringes	60% Labor				59,900
Temporary Facilities	7% Labor				7,000
Field Staff Supervision	20% Labor				20,000
SUB-TOTAL 1					1,282,300
Escalation	6% Sub-Total 1				76,900
SUB-TOTAL 2					1,359,200
Engineering and Office Costs	15% Sub-Total 2				203,900
Contractors Bond	TRW Estimate				7,500
TOTAL					1,570,600
Contractors O.H. & Profit	10% Total				157,100
TOTAL ESTIMATED PROJECT COST					1,727,700

8. APPLICATION STUDIES

8.1 APPLICABILITY OF GRAVICHEM PROCESS FOR U.S. COALS

There are presently two principal methods for the pre-combustion removal of pyritic sulfur from coal; these are the deep physical cleaning of coal (which segregates an ash and pyrite-rich coal reject from a low-ash, low-pyrite coal product) and the Gravichem Process for chemically removing pyrite from coal. Deep cleaning has been practiced for many years, primarily to produce coking coal, while the Gravichem Process is an emerging technology.

A simplified approach for comparing physical cleaning and the Gravichem Process is offered here. This method compares potential sulfur oxide emission reduction for regions of the U.S. now using high sulfur coal. The main data sources are: Steam Electric Plant Factors and Coal Data published by the National Coal Association[7,8] which provide sulfur content, in lbs of $SO_2/10^6$ Btu, for both coal resources and coal consumed and the Bureau of Mines Report of Investigation No. RI 8118,[9] which provides washability data for U.S. coals and a comparable data base for forecasting the applicability of both deep cleaning and the Gravichem Process.

The high-sulfur coal regions of the United States are the Northern Appalachian States of Maryland, Pennsylvania, Ohio and most of West Virginia; the Eastern Midwest states of Illinois, Indiana and Kentucky (west); and the Western Midwest states of Arkansas, Iowa, Kansas, Missouri and Oklahoma. Approximately 59% of all U.S. production (400×10^6 tons) in 1975 was mined in these three regions, while 66% of U.S. identified bituminous coal resources (490×10^9 tons) are located there.

The potential sulfur oxide emissions for the coal resources of these three regions averages 6.1 lbs $SO_2/10^6$ Btu (Table 23), while actual coal mined and consumed in these regions has potential emissions of

TABLE 23. POTENTIAL SULFUR EMISSIONS OF COAL TREATED BY GRAVICHEM[a] PROCESS COMPARED WITH DEEP CLEANING[b] FOR HIGH SULFUR COAL REGIONS OF U.S.

Region	Process	Potential Emissions lbs $SO_2/10^6$ Btu		% 1975[c] U.S. Prod.	% U.S. Bituminous[c] Identified Resources
		Coal Resources	Coal Consumed[d]		
N. Appalachia	-	4.8	4.3	37%	28%
	Deep Clean	3.2	-	-	-
	Gravichem	1.8	-	-	-
E. Midwest	-	6.5	5.3	22%	29%
	Deep Clean	4.4	-	-	-
	Gravichem	2.9	-	-	-
W. Midwest	-	9.0	5.9	2%	9%
	Deep Clean	6.1	-	-	-
	Gravichem	2.9	-	-	-
Three Regions	-	6.1[e]	4.9[f]	61%	66%
	Deep Clean	4.1	-	-	-
	Gravichem	2.4	-	-	-

[a] Gravichem reduction of inorganic sulfur to 0.2% w/w for coal samples as listed in Reference 10. Gravichem heat content increases of 6.3%, 3.3%, and 6.2%, respectively for N. Appalachia, E. Midwest and W. Midwest were used as reported in Reference 10.

[b] Float-sink data on seam samples crushed to 1-1/2" x 0, float all sizes at 1.60 sp. gr., Reference 10.

[c] Reference 8.

[d] Reference 7. Weighted average by quantity consumed by each state for plants 25 MW or greater. All W. Va. consumption assigned to N. Appalachia, all Ky. consumption assigned to W. Ky., no figures for Ark. and Okl. for consumption, so not used in weighted average.

[e] Weighted average based on Identified Resources.

[f] Weight average.

4.9 lbs $SO_2/10^6$ Btu. The coal consumed can be lower in potential emissions than the resource base, due to selective mining and coal cleaning, as presently practiced.

It can be seen (Table 23) that deep cleaning of coal from these regions, at 1.6 specific gravity, would give a resource base averaging 4.1 lbs $SO_2/10^6$ Btu while application of the Gravichem Process would give a processed resource base of 2.4 lbs $SO_2/10^6$ Btu. Thus, deep cleaning would have little overall effect on reducing sulfur oxide emissions, if applied to the coal reserve base. Similar conclusions can be reach on a regional basis. Sulfur emissions reduction potential of the two approaches in compared in Table 24 on a percentage basis, where it can be seen that a 45-58% reduction in sulfur oxide emissions can be forecast for application of the Gravichem Process to the resources of these three regions, while 0-26% reduction is calculable for deep cleaning. Thus, a fully-developed Gravichem Process could be very important as a sulfur oxide control strategy.

8.2 APPLICABILITY OF THE GRAVICHEM PROCESS FOR WASTE COAL RECOVERY

8.2.1 Coal Oil or Coal Water Mixtures for Boiler Retrofit

The National Energy Plan calls for greatly increased use of coal for industrial boiler use through switching of these boilers from oil to coal. However, most existing industrial boilers cannot easily switch due especially to high ash, fouling metals and sulfur in coal as compared to oil In addition, these boiler units have no coal handling facilities so a pumpable form of coal is needed. A coal fuel, particularly adaptable to the switching of oil-fired industrial boilers to coal, could be prepared by utilizing the Meyers Process ferric sulfate leach solution in a unique way. Coal tends to be partitioned in the leach solution (which has a specific gravity varying between 1.3 and 1.5 according to the concentration) into a float fraction which was exceptionally low in ash and fouling metals and nearly free of pyrite, and a sink fraction containing most of the coal pyrite and ash which can be rejected.

The light and purified float coal can then be slurried in either oil or water and stabilized in suspension to provide a fluid fuel which can

TABLE 24. SULFUR EMISSIONS REDUCTION POTENTIAL OF GRAVICHEM PROCESS COMPARED WITH DEEP CLEANING

Region	Process	Reduction of Present Sulfur Emissions[a]
N. Appalachia	Gravichem	58%
	Deep Clean	26%
E. Midwest	Gravichem	45%
	Deep Clean	17%
W. Midwest	Gravichem	51%
	Deep Clean	0

[a] $\frac{\text{Coal consumed - coal resources}}{\text{coal consumed}}$, data in Table 23.

be pumped and burned much like oil. An ideal raw material source for this "Gravifloat" fuel is waste coal.

Over 3 billion tons of high-ash fine coal exist above ground in waste gob piles and slurry ponds[11], the result of years of crushing and cleaning operations in the Eastern portion of the United States. This reserve is largely unusable as it is mainly the high ash low heat content reject from cleaning plants and is too fine for conventional heavy media cleaning. However, gravity separation at 1.3-1.4 specific gravity would float-out a uniform product containing 3-4% ash and having heat content in excess of 13000 Btu/lb.

The use of ferric sulfate leach solution as homogeneous heavy media offers a near-term technology for recovery of these resources. Most conventional heavy media cleaning devices make use of dispersed and agitated magnetite particles. This media is incapable of cleaning fine coal at low specific gravity as is required for recovery for high quality fuel from gob.

Results for an Ohio waste coal are shown in Figure 97. The removal of coal iron impurity by the acidic leach solution wash gives an additional benefit: the ash fusion temperature is raised to the level required for retrofit of non-slagging oil-fired burners. The product float coal, fed to a previously oil-fired industrial boiler as a coal-oil slurry would be the simplest near-term system for a partial switch to coal. The resulting mixture is near ideal for this purpose (Table 25), having an ash content of only 1-1/2%.

TABLE 25. GRAVIFLOAT COAL OIL SLURRY

	Coal Oil Slurries (40% Coal)	
	Gravifloat	Average Steam Coal
Heat Content	16 - 17,000 Btu/lb	15,000 Btu/lb
SO_2	1 - 2-1/2 lbs/10^6 Btu	4 lbs/10^6 Btu
Ash	1 - 1-1/2%	7%
Fouling Metals	< 1/2%	2%

8.2.2 Gravifloat Process for Recovery of High Grade Fine Coal from Slurry Ponds

A summary of Gravifloat results previously presented in Section 5, for waste slurry pond fines from three states is shown in Table 26. Gravifloat coal yield is presented in terms of heat content (Btu) recovery.

It can be seen that the Btu recovery obtainable from each waste slurry pond is large (40-78%), the sulfur content per unit heat content is reduced by 48-58% and the ash level is very low. Note that all of the ash levels can be reduced to the 3-4% level by working at specific gravities very near 1.30. The gravities utilized in these cases were selected to give a balance of high yield and quality coal rather than only to minimize ash.

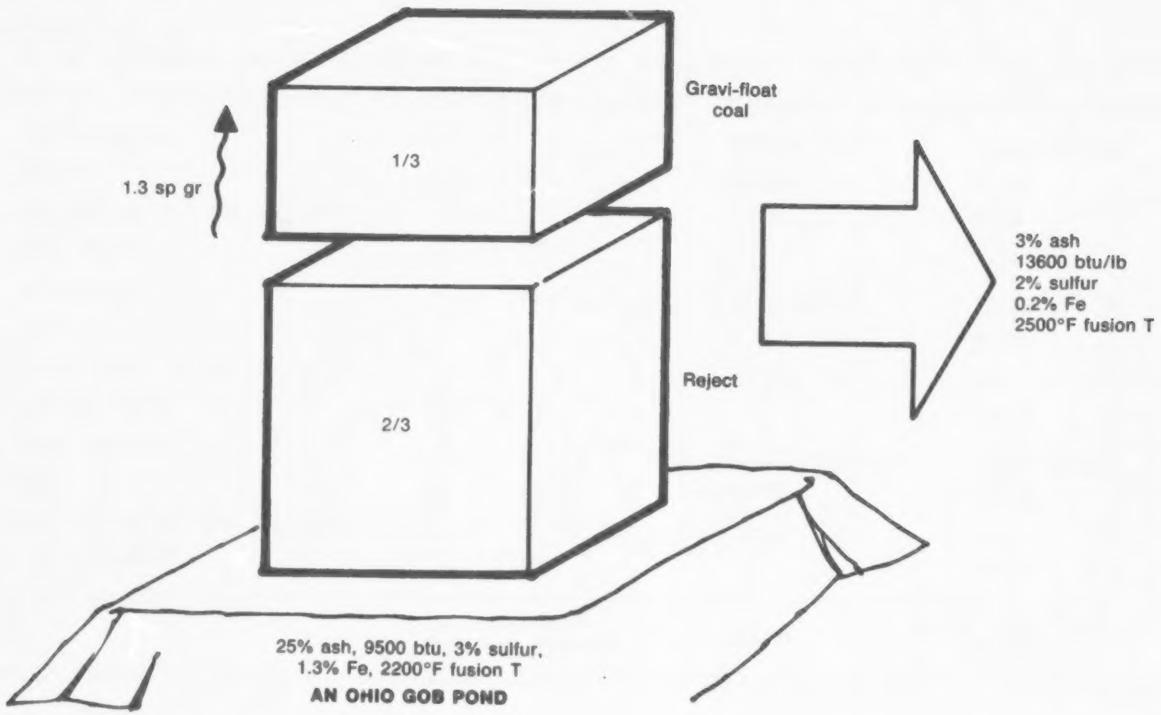
Gravi-float
coal
1.3 sp gr
1/3
2/3
Reject
3% ash
13600 btu/lb
2% sulfur
0.2% Fe
2500°F fusion T
25% ash, 9500 btu, 3% sulfur,
1.3% Fe, 2200°F fusion T
AN OHIO GOB POND

Figure 97. Gravifloat Processing of an Ohio Waste Coal.

TABLE 26. GRAVIFLOAT PROCESS RESULTS*

Refuse Location		Specific Gravity of Separation	Refuse	Product (Gravifloat Coal)	New Refuse
OHIO	Btu Recovery	1.33	-	40%	60%
	Sulfur/lbs SO_2 per 10^6 Btu		6.76	3.27	2.70
	Ash		24.88%	3.25%	28.48%
	Heat Content		9566 Btu/lb	13630 Btu/lb	9642 Btu/lb
INDIANA	Btu Recovery	1.37	-	67%	33%
	Sulfur/lbs SO_2 per 10^6 Btu		7.02	3.61	2.43%/9.08
	Ash		33.79%	8.68%	58.25%
	Heat Content		9171 Btu/lb	12922 Btu/lb	5352 Btu/lb
W.KY.	Btu Recovery	1.33	-	78%	22%
	Sulfur/lbs SO_2 per 10^6 Btu		9.39	3.93	28.13
	Ash		39.60%	5.43%	69.67%
	Heat Content		6389 Btu/lb	13529 Btu/lb	3598 Btu/lb

*Physical separation in aqueous ferric sulfate and sulfuric acid solution, e.g. 6.0% $Fe_2(SO_4)_3$ and 6.0% H_2SO_4 gives 1.33 specific gravity.

The W. Kentucky and Ohio Gravifloat coals should be particularly useful for production of low-ash coal-oil or coal-water slurries for boiler retrofit situations.

As an example, the Gravifloat separation unit could be set up at the W. Kentucky slurry pond location or one like it and operated at near 1.30 specific gravity to produce a 50% w/w float fine coal - water slurry containing coal with ash content near 3%. No final dewatering or drying step would be needed. A suspending thixotropic agent would be added so that the fine coal would not settle and the slurry could then be pumped into a tank truck and delivered for use as an oil substitute. Some modification in the oil-fired furnace would be necessary but this would be minimized by the extremely low ash of the coal. An improved particulate collection unit would also be needed.

8.2.3 Waste Coal Recovery from TVA Mine

The recovery of high grade fuel from a waste slurry pond associated with a Tennessee Valley Authority mine was pursued with some diligence, starting with selection and obtaining of the sample and continuing through evaluation of the usefulness of the results.

An excerpt of a letter to the TVA is reprinted below. This letter highlights the applicability of the subject process for a specific preparation plant waste.

The mine operator, Peabody Coal, was also contacted. Their response indicated surprise at the relatively high grade fine coal which was being ponded in apparent contradiction to the specified cleaning plant efficiency. Our impression was that they intended to try to tighten up operation of the cleaning plant rather than recover the waste. It remains to be seen whether this can be accomplished or whether the slurry pond will continue to receive the quality of coal investigated in this study.

19 March 1979

Mr. Randy Cole
Project Manager
Tennessee Valley Authority
1320 Commerce Union Bank Bldg.
Chattanooga, Tenn 37401

Dear Randy:

The sample of waste coal which you sent to us from the Camp #11 mine (Morganfield, Kentucky) was processed in our laboratories utilizing accelerated float sink separation in a water solution of iron sulfate (Gravifloat Process). The waste coal, which had a top size of less than 1/8 inch, was indeed a waste coal. It contained near 40% ash and more than 9 lbs $SO_2/10^6$ Btu. Gravifloat separation gave near 50% weight yield and a 78% Btu yield of superb float material which meets the Kentucky state standard of 4 lbs $SO_2/10^6$ Btu as shown in the table below.

	Yield				Heat Content	
	Weight	Btu	Sulfur	Ash	Btu/lb	lbs $SO_2/10^6$ Btu
Waste Coal	-	-	3.94%	39.60%	8389	9.39
Gravifloat	48%	78%	2.66%	5.43%	13529	3.93
Gravisink	52%	22%	5.06%	69.67%	3598	28.13

The sink reject contained only 22% of the waste coal heat content. Frankly, I know of no other physical cleaning technique which can perform anywhere near as well as this - so I find the results quite exciting. The accelerated float sink separation in iron sulfate solution gave very minimal retention of sulfate by either float or sink fraction. In fact, analysis showed only 0.01% sulfate retained by float and sink coal after washing.

I can envision a practical recovery of about 3/4 of heat content of fine waste coal associated with the Camp #11 mine, using the Gravifloat Process, which would provide a product coal to meet Air Pollution Control Standards for electric utilities and industrial boilers. In this latter case, gob derived Gravifloat product could be blended with oil to form a COM containing about 3% ash which would be ideal for retrofitting oil-fired industrial boilers. Alternatively, the coal could be briquetted or agglomerated for stoker fuel.

Sincerely,

Robert A. Meyers

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